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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART II: PHYSICAL PROPERTIES

by

J. P. Komorowski

National Aeronautical Establishment

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PART II: PHYSICAL PROPERTIES

EFFETS HYGROTHERMIQUES DANS LES COMPOSITES À RENFORT DE FIBRE CONTINU

PARTIE II: PROPRIÉTÉS PHYSIQUES

by/par

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W. Wallace, Head/Chef
Structures and Materials Laboratory/
Laboratoire des structures et matériaux

G.M. Lindberg
Director/Directeur

SUMMARY

This report is second in a series of literature reviews in which hygrothermal effects on aerospace composite materials (CM) with polymeric matrixes are examined. This report (Part II) deals primarily with the glass transition temperature and expansion properties due to temperature and moisture. The chapter on residual stresses is also included in this part as these stresses are a direct consequence of the expansion properties. Specific heat, thermal conductivity and emittance are briefly mentioned in Chapter 3.0, while properties connected with moisture absorption have been reviewed extensively in Part I of the series.

Other reports in this series deal with the following topics:

Part I: Thermal and Moisture Diffusion
Part III: Mechanical Properties 1
Part IV: Mechanical Properties 2
Part V: Composite Structures and Joints
Part VI: Numerical and Analytical Solutions
Part VII: Summary of Conclusions and Recommendations

A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.

RÉSUMÉ

Le présent rapport est le deuxième d'une série de dépouillements bibliographiques concernant l'influence des effets hygrothermiques sur les matériaux composites utilisés en aérospatiale. Cette Partie II porte principalement sur la température de transformation vitreuse et la dilatation sous l'effet de la chaleur et de l'humidité. Le chapitre sur les constraintes résiduaires figure dans cette partie puisqu'elles résultent directement de la dilatation. La chaleur spécifique, la conductibilité thermique et le pouvoir émissif sont traités brièvement au chapitre 3.0, tandis que les propriétés découlant de l'absorption d'humidité avaient fait l'objet d'un examen détaillé dans la Partie I de la série.

Les autres rapports de la série portent sur les sujets suivants:

Partie I: Diffusion de la chaleur et de l'humidité
Partie III: Propriétés mécaniques 1
Partie IV: Propriétés mécaniques 2
Partie V: Structures et joints composites
Partie VI: Solution numériques et analytiques
Partie VII: Résumé des conclusions et recommandations

Une liste complète des références est incluse en annexe et les nombres entre parenthèses dans le texte se rapportent à cette liste.

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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART II: PHYSICAL PROPERTIES

1.0 INTRODUCTION

The second part of the series of literature reviews* is concentrated on three properties: the glass transition temperature and expansions due to temperature and moisture. The properties connected with moisture absorption were dealt with extensively in Part I of the review.

Specific heat, thermal conductivity and emittance have not received very much attention from researchers in composites and therefore are only mentioned briefly.

Other properties may be affected by the absorption of moisture or heat but are considered of secondary importance or trivial (i.e. effect on specific weight) and are not therefore addressed in detail.

Material properties responsible for ultrasonic wave propagation were found to be affected by moisture and hygrothermal degradation. Those interested in Non Destructive Testing (NDT) of composites are referred to Kaelble and Dynes[153, 155, 95, 152], Ishai and Bar-Cohen[138, 139] and Kriz[180].

The changes in electrical properties may also be useful for NDT purposes as was shown by Cotinaud et al[68], Dewimille[87] (see Part I of review) and Kadotani[151].

The Appendix is a revised version of the bibliography for the whole series of reviews on Hygrothermal Effects on aerospace composites.

2.0 GLASS TRANSITION TEMPERATURE

At room temperature most polymer matrix materials are in a glassy state. On increasing the temperature, the elastic moduli and viscosity decrease slowly until a point is reached at which both these quantities drop drastically within a temperature range of 2° to 5°C. Discontinuity in the change of properties does not take place, however, the rates of change of various physical properties versus temperature undergo large increases. Similar effects were observed for crystals and denoted as second order transitions. However, in polymers the derivative properties do not appear to be discontinuous as in the case of crystals and to stress this difference, the use of the term glass transition temperature (Tg) is preferable to second order transition temperature[215].

The transition from a glassy to rubbery state is observed as a drastic drop in the mechanical properties of the matrix materials and is a natural upper temperature limit for structural applications of these materials. Glass transition temperature (Tg) is therefore one of the important composite material properties.

2.1 Methods of Measurements

Glass transition is manifested when a rapid rate of change of various physical and mechanical properties is observed with a slight increase in temperature. As a consequence, many methods have been used to measure the Tg. The mehods can be either static or dynamic.

---

* Only polymer matrix continuous fibre reinforced composites are considered (see Introduction to Part I).

Torsional braid analysis (TBA) is a commonly used dynamic method (Fig. 1) (ASTM2236-81). The frequency and the rate of decay of free vibrations of a pendulum are monitored with changing temperature. Transition temperature is recorded as a significant change in complex elastic shear modulus or more precisely as a maximum in the logarithmic decrement.

Static methods are more popular and generally easier. They include dilatometry, columnar loading (or linear thermal expansion method), differential thermal analysis and other methods. In dilatometry, the coefficient of cubical thermal expansion is monitored. A rather abrupt change in the value of the coefficient over a narrow temperature interval indicates the glass transition temperature.

The Tg values vary from test method to test method as the concept of second order transition is not precise. For this reason other tests were designed (i.e. Heat Distortion Temperature tests (HDT), Vicat softening temperature using a penetration probe) and are used to establish temperature envelope of polymers and for qualitative comparisons. What separates them from methods used for Tg measurement is that they do not measure any fundamental physical property. Data obtained from those tests is especially useful in applications where the factors of time, temperature, method of loading, and fiber stress are similar to those specified in that test [ASTM D648-72(78)].

Very popular tests, the heat distortion temperature (HDT) tests can have several forms (see ASTM D648-72(78) and ASTM D1637-61(76)). The HDT test, usually in the form of three point flexural displacement under constant loading, is an empirical method and does not measure any fundamental property. The temperature designated as HDT is arbitrarily chosen and therefore should not be referred to as glass transition temperature but simply as HDT. HDT tests may be useful in qualitative comparison of materials.

Tajima[283] compared results obtained using two different test methods. Materials tested were 5209 epoxy and T300/5209 composites. For dry resin, the HDT was 139°C while Tg from dilatometry was 154°C. Sykes et al[281] used TBA (Fig. 1) and HDT (Fig. 2) for the same T300/5209 composite. The difference between Tg from TBA and HDT was very pronounced for wet composites (Fig. 3).

Carter et al[52] suggested a practical method of defining a Tg value from a heat distortion experiment (flexural). Their method is demonstrated in Figure 4 and based on an equation derived from a viscoelastic model of cross-linked polymer. The curves shown represent probe displacement (δ) vs temperature (T). The point of maximum curvature (T_c, δ_c) is found by visual inspection or numerical analysis. The intersection between a line parellel to temperature axis at the distance of 3.60 δ_c and a tangent to the curve at (T_c, δ_c) gives Tg. This Tg value was in good agreement with results obtained from loaded columnar expansion (where a change in the first derivative of expansion curve is used as Tg). Carter et al designate the temperature of which this second order transition takes place as T_2 rather than Tg.

It should be mentioned here that the loads used in measuring the Tg or HDT usually have to be optimized for each material to increase sensitivity. The results of HDT or Tg measurement tests are generally repeatable to better than 2°C. Reproductivity is somewhat lower, but for worst conditions, should not exceed 10°C (ASTM D1637-61(76), D3418-82, D2236-81).

2.2 Glass Transition Temperature in Composites and Neat Resins

Browning et al[41] measured HDT of neat 3501-5 resin and AS/3501-5 composite. They do not make a distinction between HDT and Tg and refer to the measured softening point in deflection of their sample as Tg. The value of HDT for the dry resin was 177°C as opposed to 189°C for the composite. However, Tajima and Wanamaker[283] worked with 5209 resin and T300/5209 composite. For these materials, the result of HDT and Tg (dilatometry) tests were opposite to Browning's. Both the HDT and Tg of neat resin was higher then those for the composite in the dry state. These seemed to indicate that the effective cross-link density in the matrix is less than that in the neat resin. In the wet state the situation was reversed and the Tg and HDT values of wet composite were higher than those of wet resin (see Table 1).

2.3 Effect of Moisture on Glass Transition Temperature

The effect of moisture on Tg and HDT of epoxies and their composites has been studied extensively[8, 281, 41, 42, 211, 214, 220]. Typical results are shown in Figure 5. Even a small moisture content will lower the Tg of these materials considerably. Carter and Kibler[51] measured the effect of moisture on glass transition temperate for F178 polyimide resin. From Figure 6, it can be seen that the effect is similar to that observed in epoxies.

The lowering of Tg and HDT by moisture is observed regardless of the method of measurement. However, as McKague[214] pointed out, the column loading method is better suited for wet Tg measurement as the extension of the specimens core is measured. The surface is dried during the experiment and shrinks which must have an effect on bending tests.

Browning et al[41, 42] used Equation (1) suggested by Kelley and Bueche[165] to predict Tg of wet resin.

$$Tg = \frac{\alpha_p V_p Tg_p + \alpha_d (1 - V_p) Tg_d}{\alpha_p V_p + \alpha_p (1 - V_p)} \tag{1}$$

where:

α — expansion coefficient
V — volume fraction
suf_p — denotes polymer
suf_d — denotes diluent

This equation is based on free volume theory. Browning used $\alpha = \alpha_i - \alpha_g$ where α_i and α_g are the linear expansion coefficients above and below Tg respectively. Browning et al had to assume the Tg of water as 4°C. Their results correlated well with measurements of Tg (or rather HDT) for 3501-5 resin (Fig. 7).

DeIasi and Whiteside[81] modified Equation (1) by using specific volume of water values calculated from swelling measurements. This modification gave good correlation, especially for neat resins.

McKague[211, 214] observed that measured Tg temperatures from loaded column measurements are lower than those predicted by Equation (1). He pointed out that good correlation was obtained only for bending tests where drying of specimen surface affects the results. His modified equation introduces α_p as a volumetric expansion coefficient of a polymer in the glassy state. Swelling is also accounted for in his modified equation. The two models are compared in Figure 8.

Morgan and Mones[220] simplified Kelley and Bueche's equation. Since the expansion coefficient α_d 'amorphous water', was not known, it was assumed to be equal to α_p. The Tg of water was taken between 137°K and 182°K and the results compared with experimentally determined Tg for TGDDM-DDS epoxy-moisture system (see Fig. 9). The large discrepancy in results was attributed to the fact that in free volume theory, the hydrogen bonding interactions of active sites within epoxy are not accounted for. Morgan and Mones suggested that the Kelley-Bueche equation should not be used for predicting wet Tg in epoxies.

Carter and Kibler[51] proposed an entropy model for predicting glass transition temperatures in wet resins and composites. This model designates configurational entropy, rather then free volume, as the temperature dependent function that determines this transition. Arbitrary assignments for Tg and α of water are avoided and hydrogen bonding is accounted for. After some simplifying assumptions, the following Equation (2) is obtained:

$$Tg_f = Tg_o \, [1 - (R/M_s \Delta C_p) \, y(r)] \tag{2}$$

where:

$$y(r) \equiv r \ln \frac{1}{r} + (1 - r) \ln \frac{1}{1 - r}$$

$$r \equiv (M_s/M_w)f$$

$Tg_f(°K)$ — glass transition temperature of material containing f grams of water per gram of dry resin

$Tg_o(°K)$ — Tg of dry resin

M_s(g/mole) — effective formula weight of hydrogen bond site

ΔC_p(cal/g°C) — jump in specific heat due to glass transition in dry resin

R — universal gas constant

$M_w = 18$ g/mole — formula weight of water

Carter and Kibler encountered difficulties in measuring ΔC_p so a final comparison of the two models was not possible at the time. For some materials, when parameters in Equation (1) are well chosen, equally good correlation with experimental values may be obtained with both models. However, as shown in Figure 6, for some resins Equation (2) gives much better correlation. The full potential of this model will be realized when problems with ΔC_p measurement are overcome.

When studying the relationships of Tg, peak thermal spike and moisture, McKague[211] pointed out the importance of moisture distribution. In Figure 10, the two different moisture distributions shown result in the same allover moisture content. However, wet Tg may be locally exceeded leading to accelerated moisture absorption and deterioration of material (see Part I and Part III of the review).

2.4 Conclusions

1) HDT is often referred to as Tg. This should be avoided as the two temperatures may be substantially different.

2) Moisture significantly lowers the Tg and HDT of matrix resins[41,42,81,211,214,220,281].

3) HDT assessments of wet materials should not be based on flexural type tests[214].

4) The loaded column expansion method seems to be better suited for wet Tg measurement[214].

5) A study comparing tests in which a fundamental property is being measured and from which Tg may be determined would be useful. At present it is difficult to relate results obtained in various laboratories using various methods.

6) Of all the models used for predicting wet Tg of composites reviewed here, the entropy model seems most promising. However, problems with some measurements, i.e. ΔC_p will have to be overcome before it becomes practical[51].

3.0 THERMAL PROPERTIES — COEFFICIENT OF THERMAL EXPANSION

When considering thermal effects in solids, specific heat, thermal conductivity, emittance and coefficient of thermal expansion (CTE) are the most often used properties. Recently, the first three mentioned properties have not received much attention from researchers in composite materials.

The methods of measuring of these properties are fairly well established and the ASTM standards are good references in this case. However, finding published values of these properties for specific composites is not usually easy. For graphite/polyimide, notably, Campbell and Burleigh[49] conducted an extensive study of these properties and some results are shown in Figures 11, 12 and 13. Christensen presented methods of predicting values of these properties for composite materials[6 Chapter IX].

The fourth of the above mentioned properties, namely the CTE, has been extensively measured by various researchers and some of their results will be discussed below. Matrix materials generally have very different CTE from the fibers. As the composites are cured at elevated temperature, there is on cooling to the normal usage temperature, a complex stress state created due to this CTE mismatch. These residual stresses may cause loss of dimensional stability or even failure of the interface or matrix material. Another reason for the interest in the CTE of composites, is that due to the negative CTE of the graphite fibers and the positive CTE of the resin matrices, laminates with practically zero CTE, within wide range of temperatures, may be made (e.g. graphite/epoxy antennae, telescopes). Hertz[130] in Figure 14 gave good illustration of the advantage of composites for applications where stiffness, dimensional stability and weight are of primary concern.

Rogers, Yates et al published a series of articles on the linear thermal expansion of composites[243, 324, 325, 326, 327, 242]. An interferometric (Fizeau) method was used to measure the changes of CTE within a temperature range of 90°K to 400°K (500°K). A large amount of data was produced using different graphite fibers, orientations, fiber volume fractions, resin types and lay-ups. The effects of matrix curing characteristics and fiber weave types on CTE were also investigated. Materials used in these studies included HTS and HMS graphite fibers, ERLA 4617/m PDA, DLS 351/ BF_3 400 and code 69 (Fathergill & Harvey Ltd) epoxy resins. CTE of neat resins, unidirectional and multidirectional lay-ups were measured.

The results provide useful data which can be immediately applied. When these data were compared with current predicting methods, generally good correlation were achieved. However, a major drawback to the application of these theories, is the lack of precise data on the CTE of fibers as well as their transverse elastic properties. When appropriate values are assigned to these thermoelastic constants, current techniques account well for fiber volumes (v_f) in the range of 0.5 to 0.8 at room temperature. Measurements have shown that the higher the fiber content v_f — the higher is the temperature at which resin softening effects on CTE are observed. Accuracy of predictive methods for CTE of composites was improved when detailed data on the temperature dependence of CTE of the neat resin had been used.

Experience gained from the response of simple lay-ups to temperature change has enabled the behavior of progressively more complex structures to be predicted with some degree of confidence according to Yates et al[326].

Recently, Parker et al[229] have expanded data on the CTE of graphite/epoxies by testing Fibredux 914C resin both neat and reinforced with HTS fibers. The scatter for this system was higher than for those previously tested by Rogers, Yates eta al. The averaged results are, however, fairly representative for the graphite/epoxy systems. Some of these data are shown in Figures 15, 16 and Table 2.

Wang et al[303] conducted a study of CTE of Modmor II graphite in an epoxy resin (unspecified). They used static and creep tests to obtain data on the mechanical properties of fibers and epoxy separately. This data was later used to calculate CTE of laminae and the results were compared with experimental data (Fig. 17). Wang et al also made comparison between direct measurement and co-ordinate transformation (Fig. 18). For a bidirectional laminate, a comparison was made between direct measurement and lamination theory (Fig. 19). All theories used were linear elastic and generally good correlation was obtained except for high temperatures where viscoelastic properties become more pronounced. The results agree well with conclusions reached by Rogers, Yates et al. Parker et al[229] also compared theory with experiment for multidirectional laminates (Fig. 20). According to Parker, the poor correlation for 0/±45° laminates was caused by the presence of moisture in the specimens.

Cairns and Adams[45] measured the thermal expansion for 3501-6 resin and its unidirectional composites made with AS graphite and S2 glass fibers. The data were compared with results obtained from a micromechanical analysis based on finite element methods. The correlation was poor. This was, according to Cairns and Adams, mainly caused by the fact that transverse CTE of fibers was not known (a sensitivity study of the model was undertaken, however, no synergistic effects were considered).

Kabelka[150] formulated a model for predicting the CTE of fabric reinforced composites. Comparison between the model and experimental results (Fig. 21) show poor correlation. Kabelka concluded that this should improve when the viscoelastic response of matrix material is included. In a previously mentioned study, Rogers et al[242] found that for fabric reinforced graphite/epoxies:

- fiber tow densities play a critical role in governing the average values of in plane CTE of laminates,
- the crimp in reinforcing fibers is a significant factor in controlling the temperature at which resin softening effects become apparent in the out of plane thermal expansion behavior,
- laminate stacking may change the thermal expansion by as much as 100%.

Camahort et al[46] thermally cycled near zero CTE laminates. A near zero CTE lay-up was designed from results obtained on unidirectional specimens and was found to be $[0^\circ_4, \pm 40^\circ, \pm 70^\circ]_s$. Cycling was severe as samples were alternately immersed in liquid nitrogen and boiling water. Materials and corresponding results can be seen in Table 3. The CTE was measured over a range of temperatures from -1°C to 38°C. An increase of CTE was found to be caused by microcracking of the resin — the angle plies ceased to contribute and the laminate acted like a unidirectional one. The lower cure resin system marked 339 (121°C cure) performed better than the 177°C cure systems, as did the hybrid system where T300 plies made of woven graphite were added and these in effect acted as crack stoppers.

CTE of graphite/polyimide systems used by Campbell and Burleigh[48] showed scatter during temperature changes. According to these authors, this was also caused by microcracking of the resin.

Tennyson[286, 287] studied the effect of six months of temperature cycling from 24°C to 93°C in vacuum ($10^{-7} \sim 10^{-8}$ torr) on CTE. Over 30 cycles were applied during the whole test. Samples used were four ply $\pm\theta$ symmetrically balanced laminates of graphite, boron or Kevlar in an epoxy matrix. The results can be seen in Figures 22, 23 and 24. CTE predictions for laminates were calculated using unidirectional laminae properties in their respective environments. These are compared with data in Figures 25 and 26. Very good correlation was achieved. From these results Tennyson concluded that outgassing (mainly H_2O) and microcracking significantly affects CTE. Removal of moisture seems to increase CTE of $\pm\theta$ laminates (a similar observation was made by Parker[229]).

For 3501 epoxy, moisture seemed to have just the opposite effect (Adamson[4]). Dry epoxy samples had CTE equal to $8.0 \pm 0.5 \times 10^{-5}\,^\circ C^{-1}$ while a sample saturated (7.3% wt gain) at 74°C with moisture displayed CTE equal to $1.9 \pm 0.5 \times 10^{-4}\,^\circ C^{-1}$. (Adamson measured volumetric expansion.) Adamson described his observations of thermal expansion and swelling of epoxies in terms of free volume theory and postulated an equilibrium state existing between bound and unbound water in the resin. As free volume is inversely proportional to temperature, so is the volume of unbound water in the epoxy. As only bound water causes swelling it is important that CTE measurements for wet specimens allow for the time needed to reach equilibrium between bound and unbound water. Adamson found that one hour was sufficient time for this equilibrium to be reached at 25°C, however, at 1°C, 24 hours were needed.

For applications where dimensional stability is of primary importance, two methods of sealing the composites from moisture were suggested at General Dynamics (Hertz[130]). Both the bonded aluminium foil and tin-indium eutectic over a thin coat of copper proved to be effective barriers up to 1500 hours of exposure. However, besides affecting the weight of the composite (~ 0.13-0.21 g/in^2) the coatings affected the CTE. For unsealed GY70 — X30 $[(0/45/90/-45)_s]_2$ the CTE was -0.017×10^{-6} °C^{-1} ($-15° \div 24°$C) and $+0.021 \times 10^{-6}$ °C^{-1} ($24° \div 74°$C). When sealed with 0.02 mm single layer aluminium, the CTE changed to $+0.058 \times 10^{-6}$ °C^{-1} and $+0.089 \times 10^{-6}$ °C^{-1} respectively.

Useful data for CTE of composites at cryogenic temperatures can be found in References [163 and 65].

4.0 MOISTURE EXPANSION OF COMPOSITES

The absorption of moisture into the composite poses a problem of dimensional stability very similar to the one caused by thermal expansion. Current polymer matrices absorb moisture and expand (swell) as a result. Most fibers (glass, graphite, boron) do not seem to absorb moisture and restrict the matrix expansion especially in the longitudinal direction of unidirectional lamina. An internal stress state is thus created along with dimensional changes. Yet another stress state is created when expansion of a lamina is restricted by adjacent plies in a multidirectional laminate. Since moisture distribution through the thickness of a composite is generally not uniform, this type of internal stress may be present even in unidirectional laminate [Part I of the review].

By analogy to thermal effects, most theoretical prediction techniques developed for thermal expansion are applicable. Work by Marom and Cohn[206] is a good example of the applicability using analogies. Schapery's theory was used there to predict coefficients of swelling of single lamina parallel and transverse to fibers (μ_L and μ_T). Swelling of unidirectional laminae (E glass/Araldite F/HT 972 or ERL 2256 epoxy) was measured in plane every 15° from 0° to 90°. The results were compared with two predictions, one based on Schapery's theory, and the other based on measured μ_L and μ_T. In Figure 27, comparison is made between the results. Schapery's equations should give better correlation if wet properties of constituent materials (resin) are used.

Menges and Gitschner[216] also used this analogy and applied Schneiders equations. The relations obtained between swelling strains and fiber volume fraction were used to produce the graph shown as Figure 28.

In moisture expansion of polymeric composites, the most important problem is the expansion of the matrix material. Shirrel and Halpin[262], in their review paper, compared experimental data from Hertz, Browning and McKague with the theory based on the assumption of additivity of volumes:

$$V_{\text{DRY LAMINATE}} + V_{H_2O} = \text{Swollen volume}$$

Within the restrictions that the moisture distribution is uniform and the resin material isotropic, the dilatational strain is:

$$e = \frac{1}{3}\,\frac{\Delta V}{V_o}$$

In Figure 29 the correlation seems to be good and differences with respect to expansion due to moisture among various epoxies are small.

However, results from more recently published experiments do not lend themselves to interpolation using assumptions based on additivity of volumes (see Figs. 30, 31 and 32). Hahn and Kim[118] developed a micromechanics model in which the expansion of the resin takes place after the voids are filled with absorbed moisture. For each sample, there is a threshold value of moisture concentration below which no expansion takes place. Above the threshold the expansion is linear with moisture content. Hahn and Kim assumed that material behavior is elastic and that swelling strains are independent of temperature. Experiments did not quite agree with this model as some expansion could be seen below the threshold value (C_v) in Figure 31.

Adamson[4] used the concept of free volume to explain the thermal expansion and swelling of cured epoxy resins (see Review Part I, page 15). The free volume is the difference between the measured volume and the occupied volume. The occupied volume consists of mass volume plus the vibrational volume. In the first stage of moisture absorption only a fraction of absorbed water becomes bound to the epoxy network and causes swelling. The rest of the absorbed moisture occupies the free volume. The second stage of moisture absorption begins when the free volume is completely filled with water and all absorbed water becomes bound causing swelling. In this second stage, swelling is equal to the volume of absorbed water. In the third stage of swelling observed by Adamson water occupies free volume contained by micelles (highly cross-linked dense centers of generally less dense resin) and swelling again is less than the volume of absorbed water. As the equilibrium between bound and unbound water is inversely proportional to temperature (free volume changes with temperature, Fig. 33), swelling is also dependent on temperature. For a given amount of water in the sample, swelling will increase with an increase in temperature. With changes in temperature, it is very difficult to separate the effect of swelling from the pure thermal expansion in an experiment.

5.0 CONCLUSIONS (Chapter 3.0 and 4.0)

1) There is a lack of data on the transverse properties of fibers which in turn limits usefulness of theoretical predictions of CTE of composites.

2) Accuracy of predictive techniques for CTE of laminae are improved when the dependence on temperature of the fiber properties and even more so, the resin matrix are included in the analysis.

3) CTE of laminates can be predicted if physical properties of unidirectional laminae are known.

4) The hydrothermal history of the laminate may have an effect on its CTE, (i.e. thermal cycling which results in the microcracking of the resin matrix).

5) Moisture in the specimen complicates the measurement of CTE and in general, wet specimens require longer time at temperature before any change in dimension is measured. Wet composites have different CTE than the dry ones.

6) Even very thin layers of coating (aluminium or other metal coating which serves as moisture barrier) changes CTE of laminates significantly.

7) Swelling is significant in current polymeric composites. It can be predicted with the same expressions used for predicting the CTE. These predictions are improved when the wet properties of resins are used.

8) Resin swelling seems to be a three stage process that can be best explained with a free volume concept.

6.0 RESIDUAL STRESSES

The thermal and moisture expansion properties of fibers and matrices are such that internal stress states are present in composites under general conditions. These residual stresses may be responsible for both defects and dimensional instabilities of cured composite materials before any external load is applied.

6.1 Micromechanics

As described in previous chapters, fibers and resins have greatly varying expansion coefficients. The cure temperature of resins which are of interest here, is typically between 120°C and 180°C. Immediately after cure and before cool down, the resin is in a leathery state and any stresses created at this stage will be relaxed almost instantly. During the cool down to room temperature, the resin becomes more elastic and, owing to the mismatch of expansion coefficients and restraints provided by the fibers, an internal stress state will result. This type of stress was studied in model materials made of thick glass filaments embedded in birefringent resins (epoxies typically are birefringent materials). Recently, Cunningham et al[75] used this method and obtained photoelastic data using a polarizing microscope. The principal stresses were obtained with an oblique incidence method. A plot of loop stress around the fiber is shown in Figure 34. The maximum loop stresses at the surface of the fiber were calculated to be approximately 100 MPa (tensile) while radial stresses were only 6MPa (compression).

Adams and Miller[2, 218] conducted finite element inelastic analyses of hygrothermal microstresses and their results generally agree well with Cunningham's photoelastic investigation. The magnitude of these microstresses was sufficient to cause yielding of the matrix. Hygrothermal cycling was shown to change the state of residual microstresses with subsequent dimensional instability of laminae.

Menges and Gitschner[216] demonstrated yet another mechanism for the rise of residual stresses in the transverse direction. In this direction, resin swelling is hardly restrained by the fibers, yet stresses still build up. Fiber distribution may be homogeneous or inhomogeneous (see Fig. 35). For both types of distributions zones of high and low fiber content may be identified and these will have different swelling strains. As a result, local residual stresses are present, and as Menges and Gitschner estimated, may lead to microcracking of the resin in a glass/resin composite.

6.2 Macromechanics

Residual stresses have been observed in composite materials not only at the microscopic level, but also at the macroscopic level in angle plied laminates or in unidirectional hybrids. These stresses have been studied experimentally and analytically by numerous authors.

Chamis[53] provides a good review of NASA Lewis Research Center activities and of NASA sponsored research in this field. He pointed to four factors from which lamination residual stresses (LRS) originate:

1) the differences in the ply CTE in the longitudinal (α_{11}) and in the transverse (α_{22}) direction. $\alpha_{22}/\alpha_{11} \cong 20$ for fiber/resin laminae;

2) the difference in ply orientation angle (θ) — there are no LRS in unidirectional laminates;

3) the difference between fabrication temperature and use temperature;

4) the ability of a ply to support stresses along its material axes.

Water is present in laminates under general usage conditions which implies, from the results of the previous chapter and from literature which will be reviewed below, that point one above should be broadened to include the difference in moisture expansion, as well as CTE. However, general points brought forward by Chamis are still true owing to the analogy between moisture and thermal effects.

The existence of LRS is evidenced by transply cracks and warpage of unsymmetric angle-plied laminates before any mechanical load was applied to them. The transply cracks are observed when LRS exceed the ply transverse strength (typically 28-63 MPa)[53]. Molcho and Ishai[219] observed these cracks in a graphite/epoxy T300/5208 skin for a control surface. The original lay-up contained 0° and ±45° plies and after curing, not only transverse but also interlaminar cracks were observed. The latter originated from the tips of transverse cracks. It is worth noting that they found a uniform distribution of plies, as opposed to a lamped lay-up, to be preferable, as it resulted in less severe cracks.

Strain gauges have been employed in LRS measurements. Daniel, Liber and Chamis[78] embedded strain gauges during fabrication of laminates. Results for 0° plies of baron/epoxy $[0_2/\pm 45°]_s$ laminate are shown in Figure 36.

Pagano and Hahn[228] used a simplier method of LRS measurement. They bonded gauges to laminates at RT and then heated them to cure temperature.

The most common method of measuring LRS is through deformation measurements of unsymmetric laminates. Unsymmetric laminates warp due to the existence of LRS. This method is however indirect as it is necessary to use lamination theory to obtain stress values corresponding to the measured warpage.

Chamis in his review reported on use of linear elastic laminate theory for ply thermal stresses (LRS caused by cure temperature being different then the use temperature). Effects of some material variables were analyzed with the aid of this model. In Figure 37(c) the effect of the lamping of plies in angle-plied laminate is shown and correlates well with the earlier mentioned findings of Molcho and Ishai. The effect of fiber volume ply angle, and matrix modulus is shown in Figures 37(a), (b) and 38.

Lee, Lewis and Sacher[188, 189] used [90°/0°/90°] glass/epoxy systems to study the effect of moisture on their mechanical properties. First ply failure, in the tension test, results in a change in slope of the stress-strain curve. Lee et al called this the yield point. They observed that yield strain was affected by residual strain (Fig. 39) and these in turn changed with the amount of moisture in the composite. They measured residual stresses in symmetric laminate by the deply technique. When one of the plies was removed, the laminate wareped and the stresses could be calculated.

Pagano and Hahn[228], Hahn[119] and Hahn and Kim[118] developed a linear elastic model to analyze curing stresses, however, they used a temperature dependent elastic moduli. Measurements of warpage in unsymmetric angle plied laminates were compared with predictions. The stress free temperature (T_0) was observed to be below the curing temperature. For T300/5208 and Scotchply 1002 (glass/epoxy) it was 121°C as opposed to a cure temperature of 177°C. In transverse direction it was found that the curing stresses may be more than 50% of UTS in this direction. For swelling, a moisture threshold model was used and it was found that moisture obsorption may result in a stress free laminate at room temperature (Fig. 40). Correlation of experimental data with calculated values was not alwasys satisfactory.

The elastic model does not account for significant stress relaxation, especially near the Tg of the resin. The lowering of the stress free temperature in a composite due to annealing, can readily be explained on the basis of a viscoelastic model. Crossman, Warren and Pinoli[73] studied the relations between residual stresses and annealing time and temperature (see Fig. 41). For Shell 1031 resin (190°C cure) the residual stresses at room temperature were lowered by 25% or ~ 7 MPa after 16 hours at 121°C. Moisture, it was concluded, by plasticizing the resin, further accelerates residual stress relaxation. Crossman and Flaggs[74, 101] compared results of a linear viscoelastic analysis of dimensional changes in laminates with experimental data and the results of elastic analysis. The correlation between viscoelastic analysis and experimental data (warping) was very good. The elastic model failed especially when used to analyze 121°C cure material (GY70/CE339). The viscoelastic model supported earlier observations (i.e. Crossman, Mauri and Warren[69]) that the viscoelastic stress relaxation during complete moisture absorption-desorption cycle at constant temperature can lead to unrecoverable dimensional changes and result in different residual stress state. Thus accelerated conditioning programs may result in a changed residual stress state. Crossman et al[69] found that hygrothermal cycling increased residual stresses in a T300/5209 unsymmetric laminate. Similar laminates made from T300/5208 showed a 5-10% loss of residual stress under the same thermal history.

Hedrick and Whiteside[128] demonstrated that choosing an appropriate cooling rate for graphite/epoxy laminates (based on 3501 epoxy), may also reduce the residual stresses through relaxation. Stress relaxation was studied (Fig. 42) and different cooling rates from 177°C to 121°C were investigated. In Table 4, a summary of photomicrographic examinations is given.

Harper and Weitsman[124] and Douglas and Weitsman[94] postulated that if residual stress relaxation is possible, than there should be an optimum cool down path for a laminate. They used a linear viscoelastic model to find this path. Experimental confirmation of their finding has not been very successful so far, as specimens used were relatively thick and contained many transverse cracks which obscureed the results (an AS-3502 system was used). Further experiments in this optimal time-temperature path are continuing.

6.3 Conclusions

1) The mismatch of thermal and moisture expansion of constituent materials or individual plies in a laminate is responsible for build up of residual stress states. Residual stresses are usually significant under normal usage conditions.

2) Residual stresses (or cure stresses) may cause transverse and interlaminar cracks in laminates before any external load has been applied. These stresses may be reduced by taking advantage of stress relaxation techniques.

3) Viscoelastic models seem to be better suited for studying residual stresses in composites.

4) When accelerated hygrothermal conditioning programs are used, care should be taken, as a residual stress state may result which would not be found under normal service conditions.

TABLE 1

GLASS TEMPERATURE[283]

Sample	M_s g/100g	HDT(°C) Penetration	Tg(°C) Dilatometry
5209	Dry	134	154
	3.730	95	90
6-Ply	Dry	109	131
	1.236	90	109
12-Ply	Dry	116	131
	1.329	96	109

TABLE 2

SMOOTHED VALUES OF THE LINEAR THERMAL EXPANSION COEFFICIENT α OF THE SPECIMENS AT THREE REPRESENTATIVE TEMPERATURES[229]

Fibre Lay-Up*	Fibre Volume Fraction (%)	Area of Plate Containing More Than 1% Voids (%)	Area of Plate Containing More Than 2% Voids (%)	$\alpha(^\circ C^{-1})$ at $-150^\circ C$	$\alpha(^\circ C^{-1})$ at $0^\circ C$	$\alpha(^\circ C^{-1})$ at $150^\circ C$
100%/0°	67.1	0	0	-0.81×10^{-7}	-1.69×10^{-7}	-1.47×10^{-7}
75%/0°, 25%/±45°	65.8	0	0	-2.95×10^{-7}	-4.29×10^{-7}	-0.27×10^{-7}
50%/0°, 50%/±45°	68.6	5	0	1.40×10^{-7}	0.22×10^{-7}	4.72×10^{-7}
25%/0°, 75%/±45°	69.3	5	0	6.90×10^{-7}	7.85×10^{-7}	1.22×10^{-6}
50%/0°, 50%/90°	65.1	0	0	1.75×10^{-6}	1.88×10^{-6}	2.96×10^{-6}
25%/90°, 75%/±45°	69.3	5	0	3.65×10^{-6}	4.38×10^{-6}	5.15×10^{-6}
50%/90°, 50%/±45°	68.6	5	0	5.94×10^{-6}	7.98×10^{-6}	1.10×10^{-5}
75%/90°, 25%/±45°	65.8	0	0	8.55×10^{-6}	1.26×10^{-5}	1.75×10^{-5}
100%/90°	67.1	0	0	1.56×10^{-5}	2.41×10^{-5}	3.03×10^{-5}
No fibers				2.68×10^{-5}	4.45×10^{-5}	6.76×10^{-5}

* In relation to the direction of measurement

TABLE 3

THERMAL CYCLING EFFECTS ON LONGITUDINAL CTE[46]

Material System	Fiber Volume, %	Longitudinal CTE, μin./in.-°F	
		As-Cured	Thermally Cycled
HMS/934, U/D	65.6	−0.45	−0.35
HMS/3501, U/D	65.2	−0.35	−0.38
HMS/934, O/D	65.1	−0.10	−0.30
HMS/3501, O/D	69.5	−0.13	−0.26
HMS/339, O/D	60.6	−0.10	−0.12
Hybrid (HMS/3501 + T-300/934)	63.3	−0.13	−0.25

Note: U/D = unidirectional; O/D = oriented.

TABLE 4

SUMMARY OF PHOTOMICROGRAPHIC EXAMINATION OF Gr/Ep LAMINATES[128]

Cooling Rate (350°F to 250°F), °F/min.	No. Specimens Examined	Total Area* Examined, in.^{2}	Total No. of Cracks	Max. Crack Length, in.
5 to 6	3	3.24	47	0.012
2.5	2	1.89	3	0.005
1	9	7.21	0	—

* Microscopic examination of sectioned sine wave spars and travelers (300 X to 500 X)

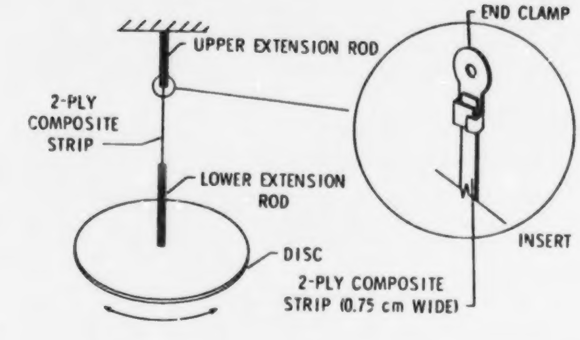

END CLAMP
UPPER EXTENSION ROD
2-PLY COMPOSITE STRIP
LOWER EXTENSION ROD
DISC
INSERT
2-PLY COMPOSITE STRIP (0.75 cm WIDE)

FIG. 1: SKETCH OF THE TBA PENDULUM. INSERT SHOWS METHOD USED TO ATTACH END CLAMPS TO COMPOSITE SPECIMEN[281]

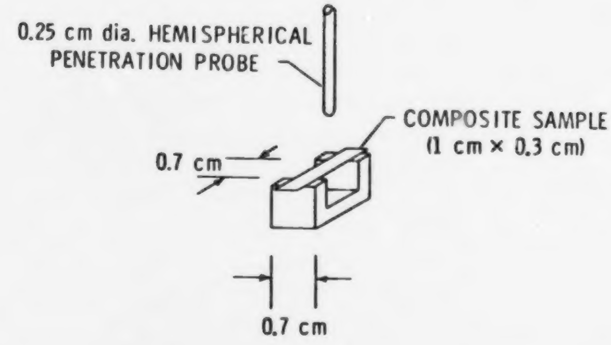

0.25 cm dia. HEMISPHERICAL PENETRATION PROBE
COMPOSITE SAMPLE (1 cm × 0.3 cm)
0.7 cm
0.7 cm

FIG. 2: SCHEMATIC OF THE TMA SAMPLE SUPPORT FIXTURE USED TO DETERMINE HEAT DISTORTION TEMPERATURES IN COMPOSITE MATERIALS[281]

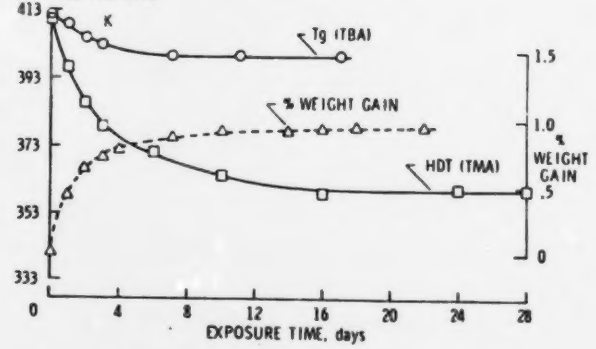


FIG. 3: EFFECT OF SOAK TIME ON THE HDT, Tg AND MOISTURE PICK UP OF A 2-PLY UNIDIRECTIONAL T-300/5209 COMPOSITE CURED AT 400 K[281]

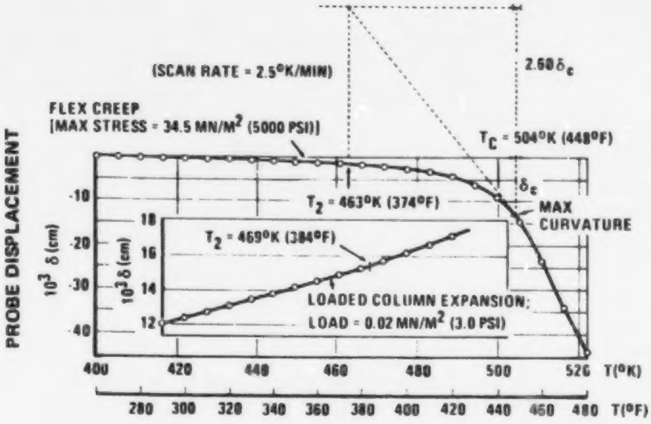


FIG. 4: TMA FLEXURE AND COLUMN LOADING RESULTS FOR DRY 5208 RESIN[52]

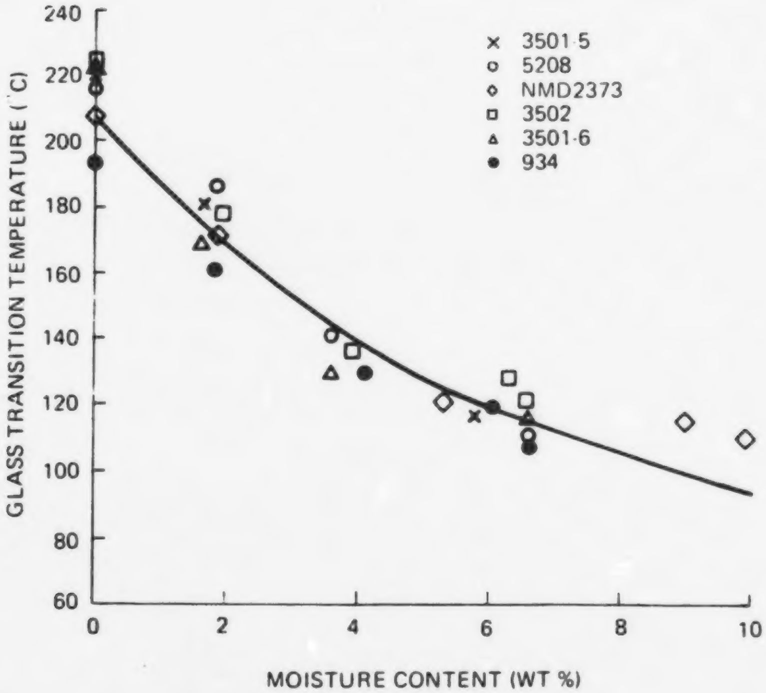


FIG. 5: RELATIONSHIP BETWEEN EQUILIBRIUM MOISTURE CONCENTRATION AND GLASS TRANSITION TEMPERATURE OF EPOXY RESINS[81]

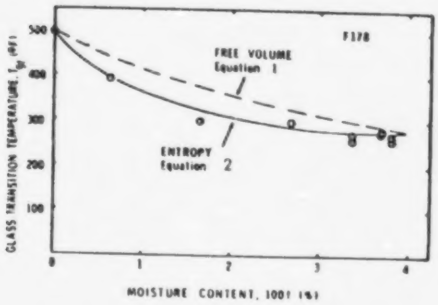


FIG. 6: VARIATION OF GLASS TRANSITION TEMPERATURE WITH MOISTURE CONTENT FOR CURED F178 POLYIMIDE RESIN. SOLID CURVE IS PREDICTION OF ENTROPY MODEL, EQUATION 2, USING $T_{go} = 533°K$ AND ARBIRARY PARAMETERS $M_s = 270$ g/mole, $\Delta C_p = 0.022$ cal/g°C. DASHED CURVE IS PREDICTION OF FREE VOLUME THEORY, EQUATION 1, WITH $T_{gp} = 533°K$, $\alpha_p = 1.98 \times 10^{-4} °C^{-1}$, $T_{gd} = 4°C$, $\alpha_d = 3.66 \times 10^{-3} °C^{-1}$[51]

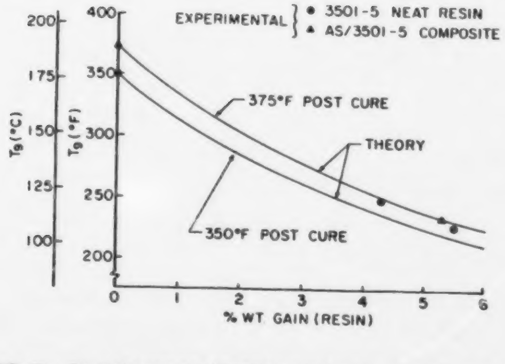


FIG. 7: GLASS TRANSITION TEMPERATURE AS A FUNCTION OF ABSORBED MOISTURE IN RESIN[41]

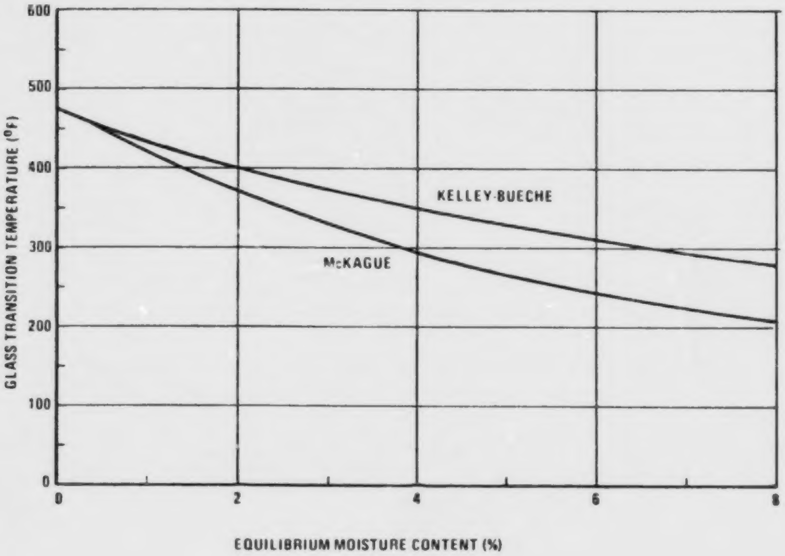


FIG. 8: COMPARISON OF GLASS TRANSITION MODELS[214]

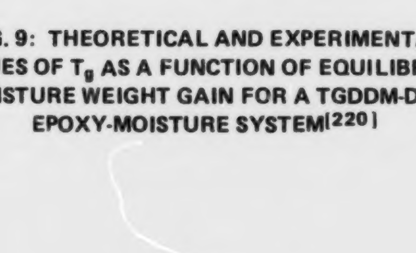


FIG. 9: THEORETICAL AND EXPERIMENTAL VALUES OF T_g AS A FUNCTION OF EQUILIBRIUM MOISTURE WEIGHT GAIN FOR A TGDDM-DDS EPOXY-MOISTURE SYSTEM[220]

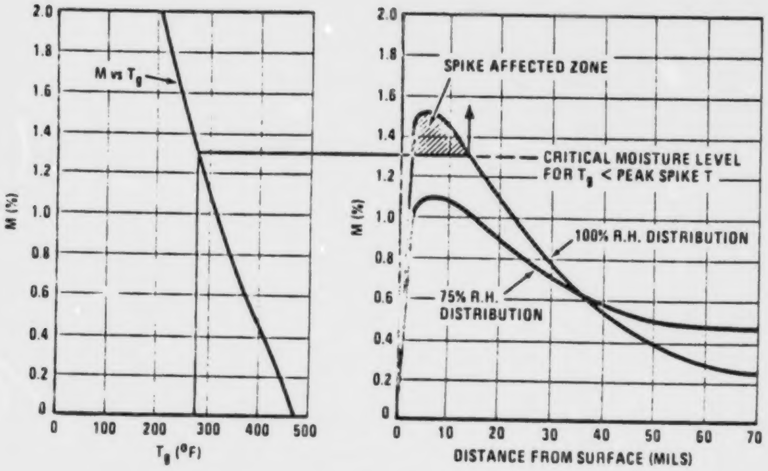


FIG. 10: RELATIONSHIP OF T_g, PEAK SPIKE TEMPERATURE AND MOISTURE DISTRIBUTION TO THE THERMAL SPIKE EFFECT[211]

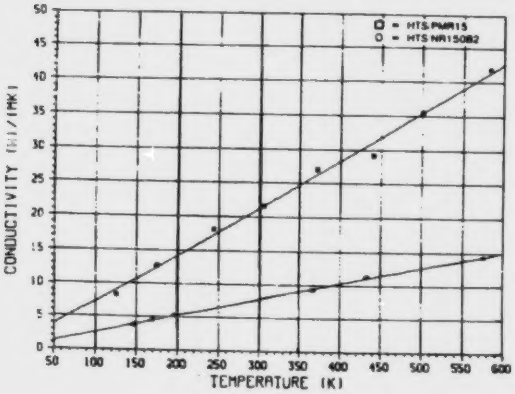


FIG. 11: THERMAL CONDUCTIVITY, $[0°]_n$ COMPARISON (CUT BAR)[48]

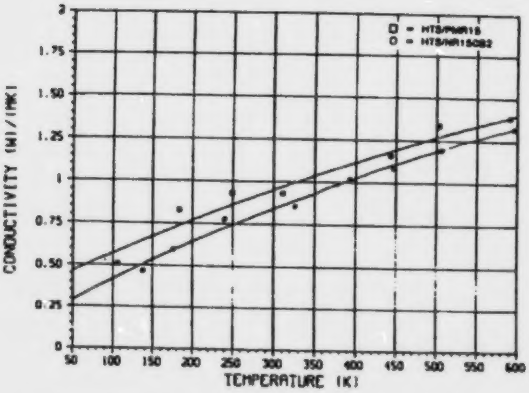


FIG. 12: THERMAL CONDUCTIVITY, $[90°]_n$ COMPARISON (HOT PLATE)[48]

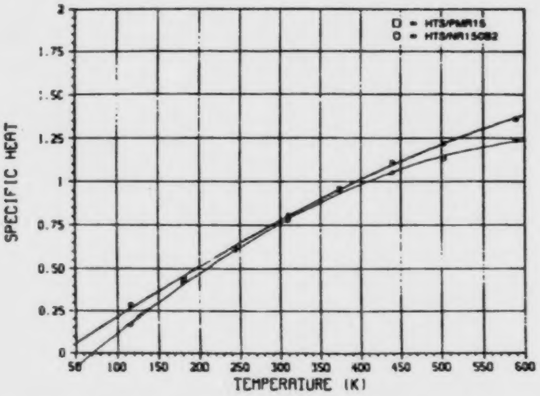


FIG. 13: SPECIFIC HEAT COMPARISON ISOTROPIC LAY-UP[48]

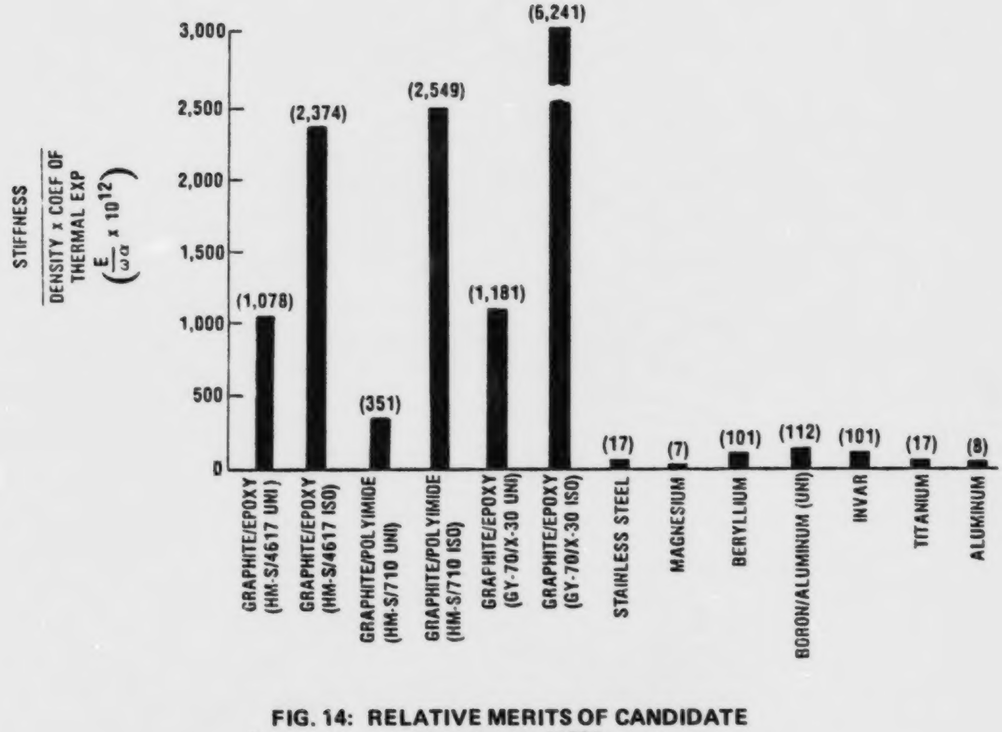
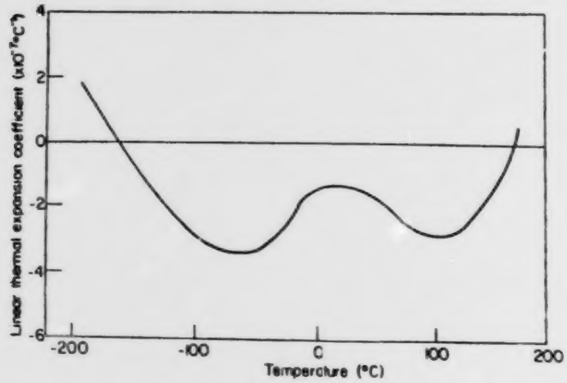
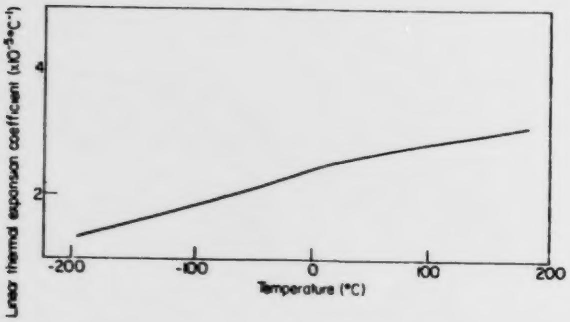


FIG. 14: RELATIVE MERITS OF CANDIDATE MATERIALS[130]



(a)



(b)

FIG. 15: THE LINEAR THERMAL EXPANSION COEFFICIENT OF $[0°]_{16}$ LAMINATE IN (a) LONGITUDINAL AND (b) TRANSVERSE DIRECTION[229]

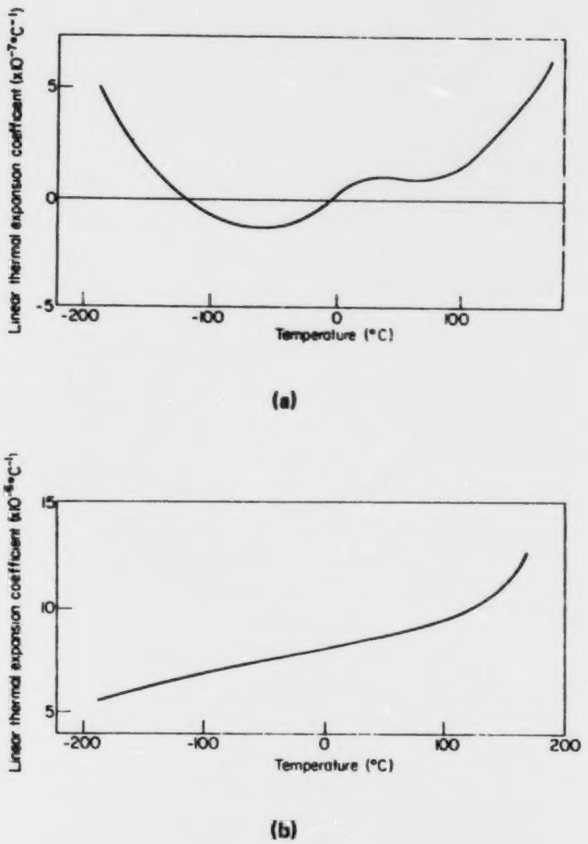


FIG. 16: THE LINEAR THERMAL EXPANSION OF $[+45°, 0°, -45°, 0°]_{2s}$ LAMINATE IN (a) LONGITUDINAL AND (b) TRANSVERSE DIRECTION[229]

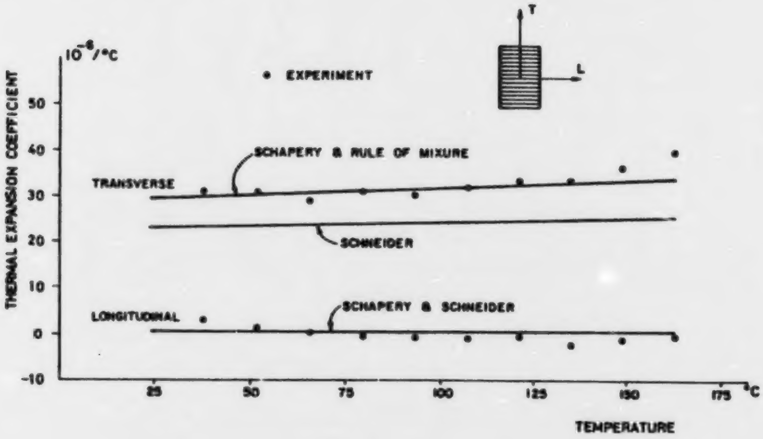


FIG. 17: COMPARISON OF THE CTE BETWEEN EXPERIMENT AND THEORY[303]

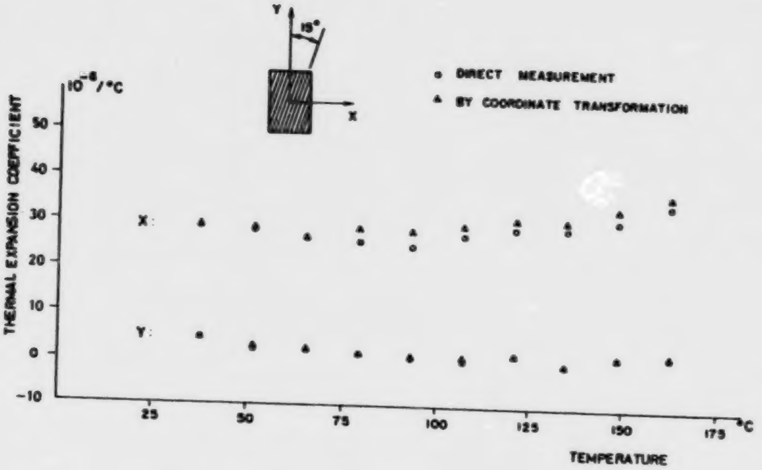


FIG. 18: COMPARISON OF THE CTE BETWEEN DIRECT MEASUREMENT AND COORDINATE TRANSFORMATION[303]

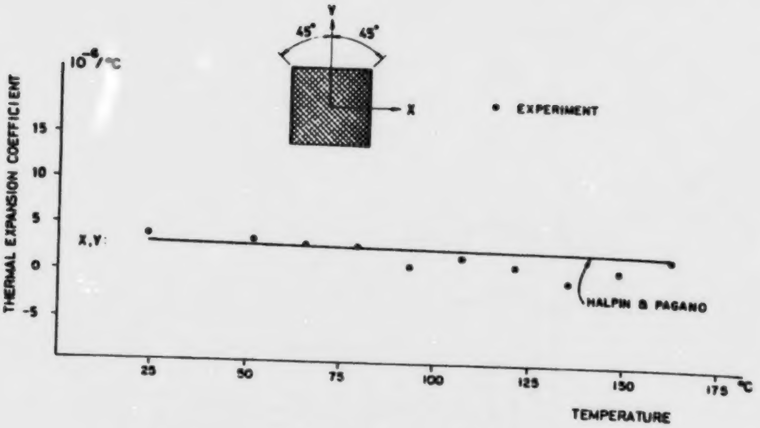
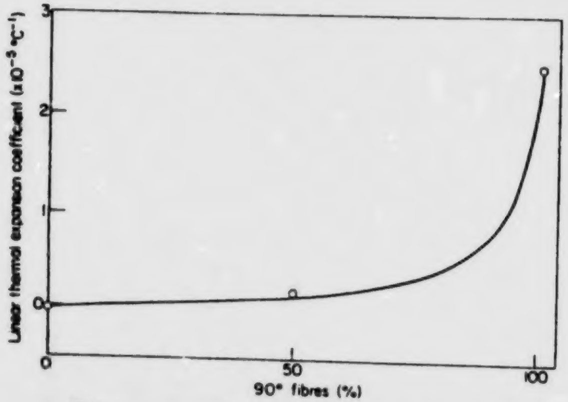
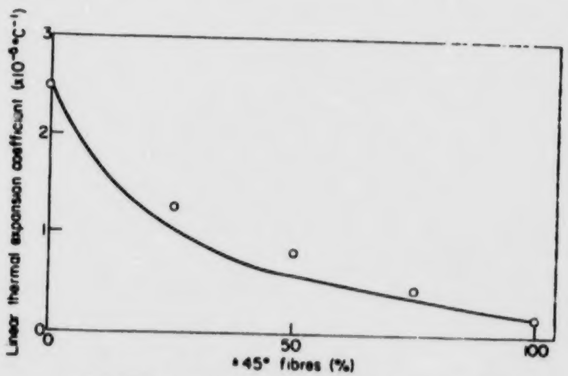


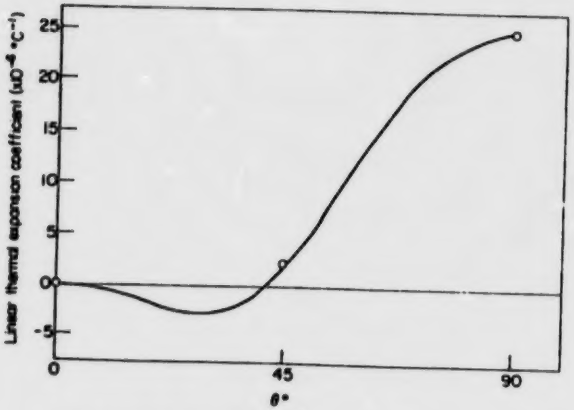
FIG. 19: COMPARISON OF THE CTE FOR A ±45-DEG LAMINATE BETWEEN EXPERIMENT AND THEORY[303]



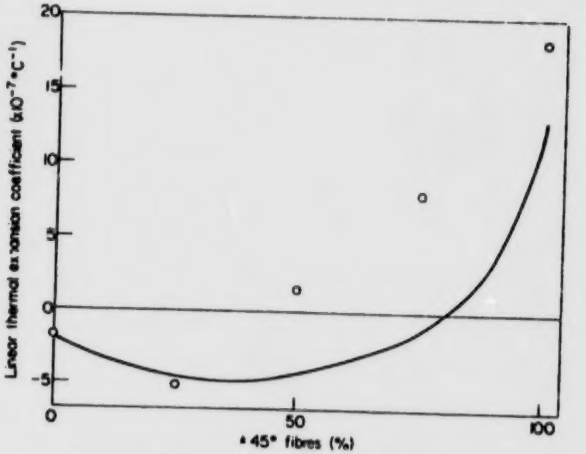
The room temperature linear thermal expansion coefficients of 0°, 90° laminates consisting of Fibredux 914C resin containing Courtaulds HTS carbon fibre present at 66% by volume: o – experimental; solid line – calculated



The room temperature linear thermal expansion coefficients of 90°, ± 45° laminates consisting of Fibredux 914C resin containing Courtaulds HTS carbon fibre present at 66% by volume: o – experimental; solid line – calculated



The room temperature linear thermal expansion coefficients of symmetrically balanced angle-ply (± θ) laminates consisting of Fibredux 914C resin containing Courtaulds HTS carbon fibre present at 66% by volume: o – experimental; solid line – calculated



The room temperature linear thermal expansion coefficients of 0°, ± 45° laminates consisting of Fibredux 914C resin containing Courtaulds HTS carbon fibre present at 66% by volume: o – experimental; solid line – calculated

FIG. 20: THE ROOM TEMPERATURE LINEAR CTE'S[229]

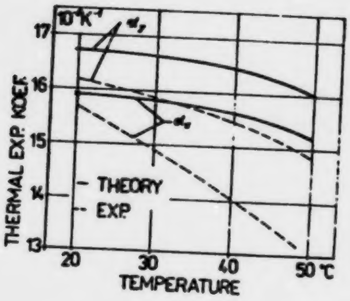


FIG. 21: COMPARISON OF THE THEORETICAL AND EXPERIMENTAL RESULTS[150]

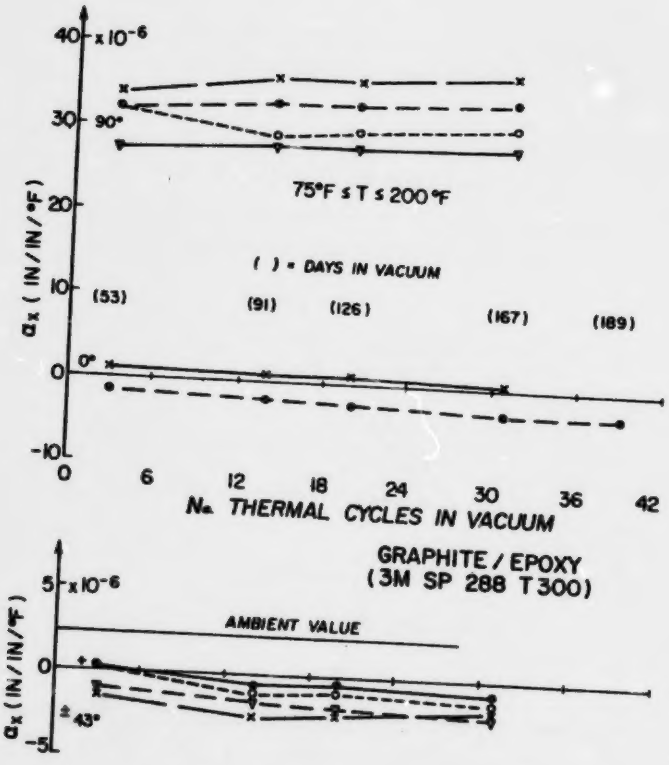


FIG. 22: THE EFFECT OF THERMAL CYCLING IN VACUUM ON CTE OF GRAPHITE/EPOXY[286]

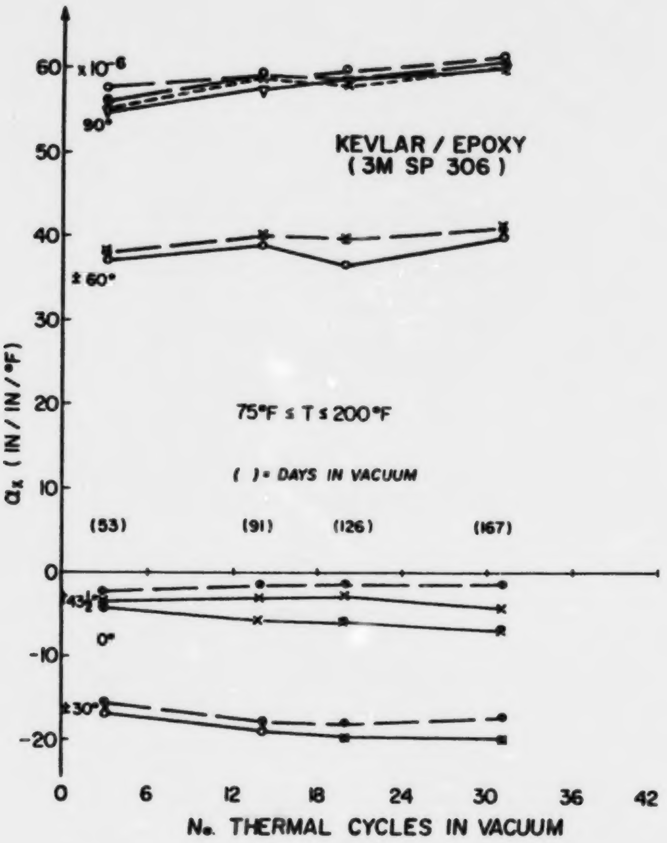


FIG. 23: THE EFFECT OF THERMAL CYCLING IN VACUUM ON CTE OF KEVLAR/EPOXY[286]

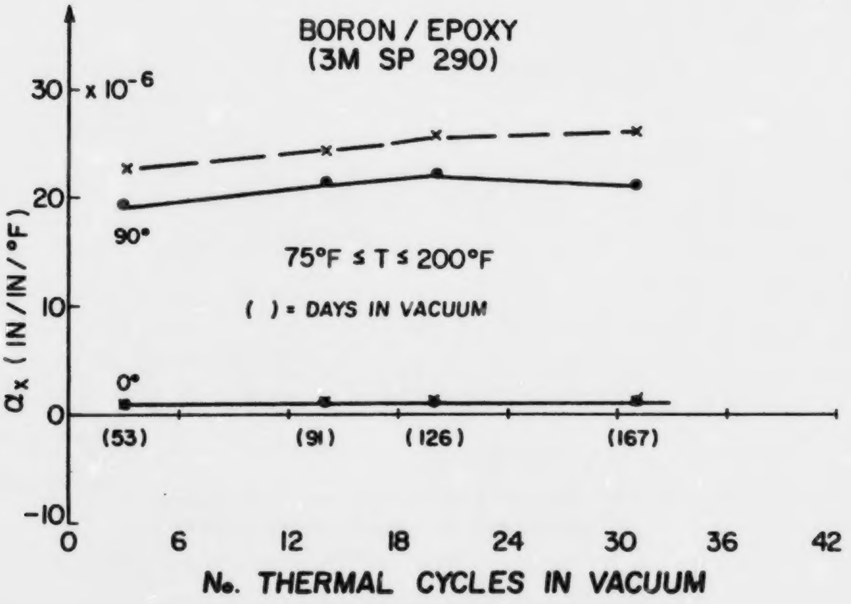


FIG. 24: THE EFFECT OF THERMAL CYCLING IN VACUUM ON BORON/EPOXY[286]

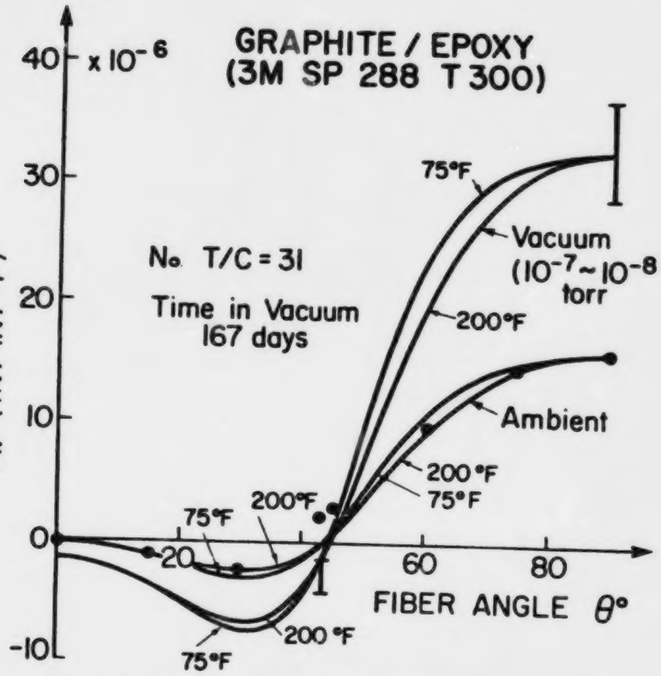


FIG. 25: COMPARISON OF CTE PREDICTED FOR VARIOUS θ WITH THE DATA[286]

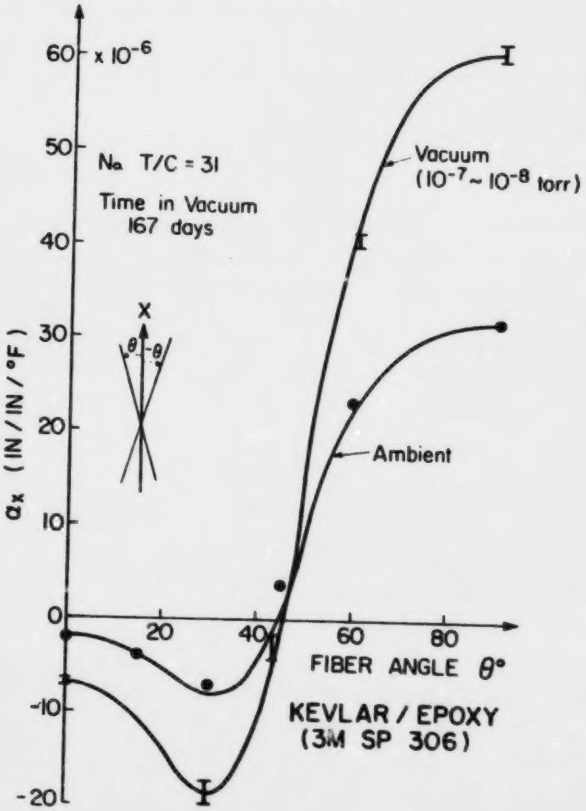


FIG. 26: COMPARISON OF CTE PREDICTED FOR VARIOUS θ WITH THE DATA[286]

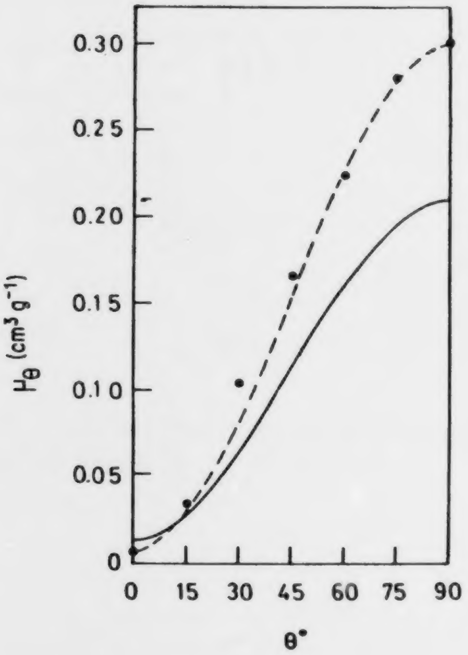


FIG. 27: COEFFICIENTS OF HYGROCLASTICITY AS FUNCTIONS OF θ, COMPARED WITH THE CALCULATION BASED ON EXPERIMENTAL μ_L AND μ_T (- - -), AND ON μ_L AND μ_T WORKED OUT BY SCHAPERY'S EQUATIONS TAKING THE CONSTITUENTS' ORIGINAL PROPERTIES (——).[206]

● MEASURED

$$\mu = \frac{\Delta L/L_o}{\Delta\omega/V_o}$$

L_o = length of specimen

$\Delta\omega$ = weight of water absorbed

V_o = original volume

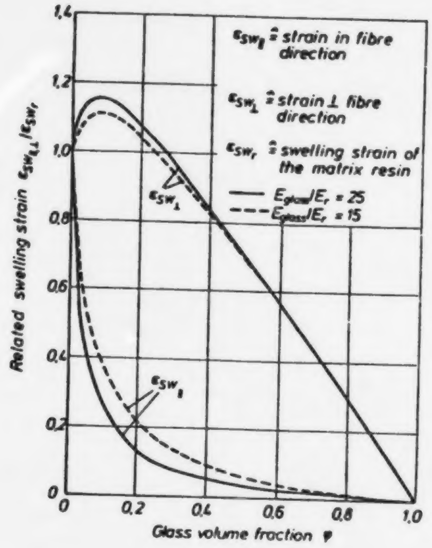


FIG. 28: SWELLING STRAIN BEHAVIOR OF ANISOTROPICALLY REINFORCED INDIVIDUAL LAYERS DUE TO MATRIX SWELLING[216]

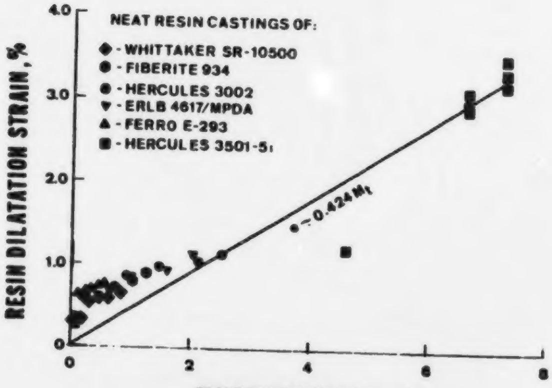


FIG. 29: SWELLING BEHAVIOR IS SIMILAR FOR SEVERAL EPOXIES[262]

RESIN	NO. DATA POINTS	LEAST SQUARES FIT	
		SLOPE	INTERCEPT
2373	51	0.840	−0.511
3501-5	42	0.977	−0.457
5208	34	0.765	−0.216

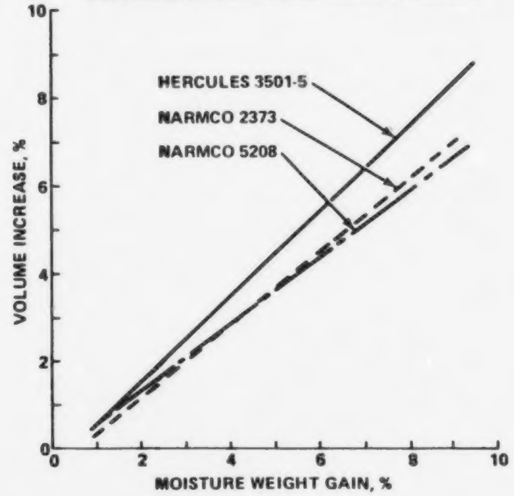


FIG. 30: RESIN SWELLING DUE TO ABSORBED MOISTURE[128]

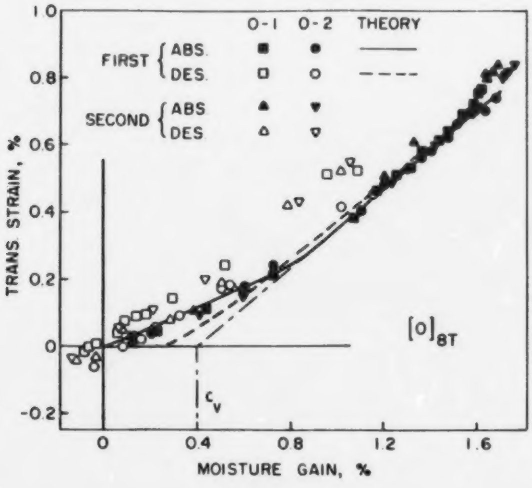


FIG. 31: TRANSVERSE SWELLING STRAIN OF $[0]_{8T}$ LAMINATE (AS/3501-5)[118]

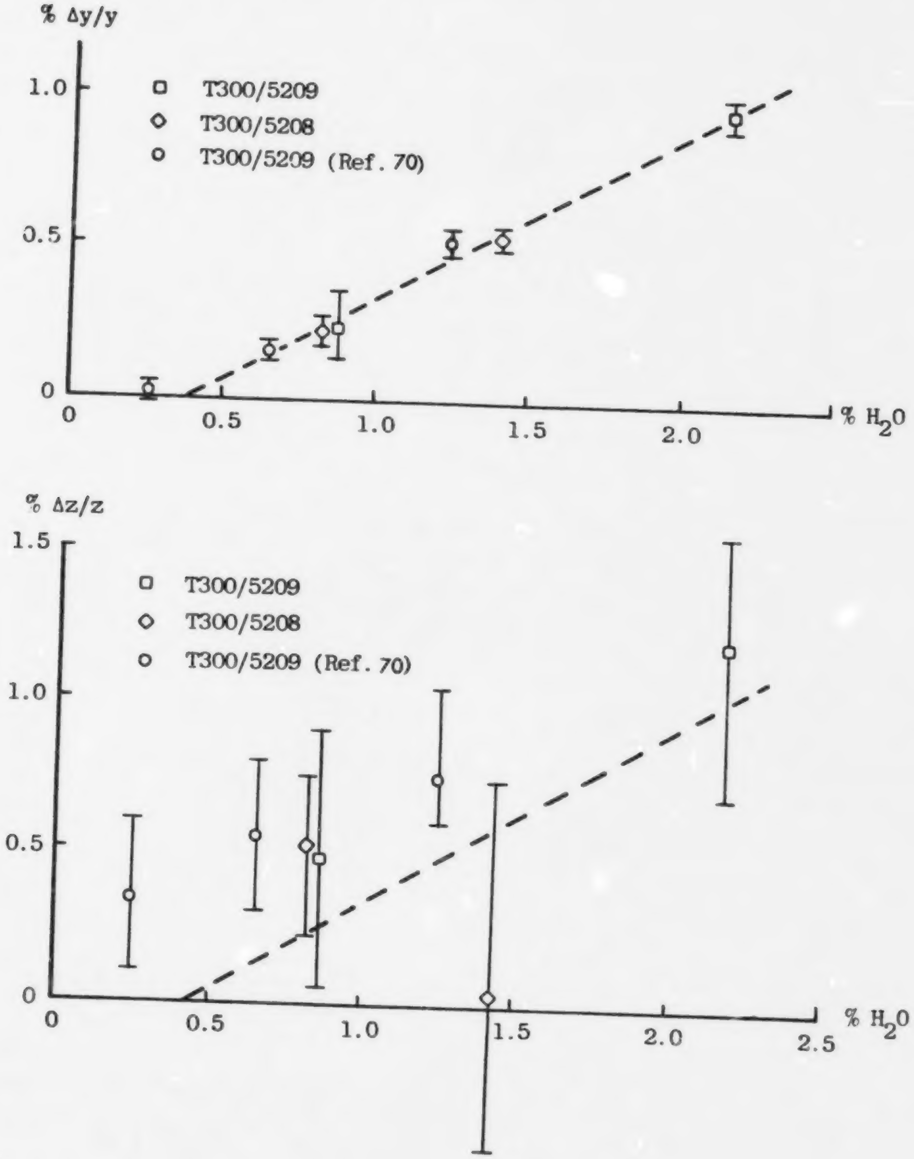


FIG. 32: DIMENSIONAL CHANGES IN TRANSVERSE (Δy/y) AND THROUGH THICKNESS (Δz/z) DIRECTIONS IN UNIDIRECTIONAL COMPOSITES vs EQUILIBRIUM MOISUTRE CONTENT[69]

The dotted line represents linear regression on Δy/y data for both graphs (Δz/z seems to be greater than Δy/y)

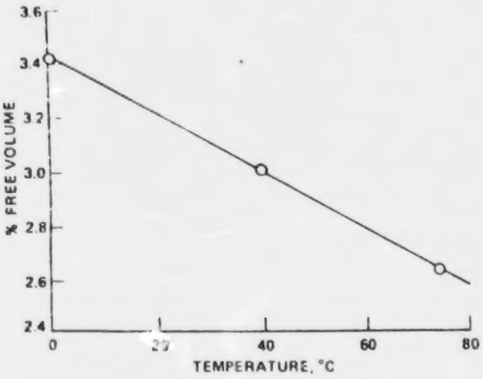


FIG. 33: PERCENT FREE VOLUME AS A FUNCTION OF TEMPERATURE, HERCULES 3501 RESIN. VALUES OF 74 AND 40°C DETERMINED BY SWELLING DATA DURING ABSORPTION. VALUE AT 1°C DETERMINED USING REVERSE THERMAL EFFECT FROM 74°C EQUILIBRIUM[4]

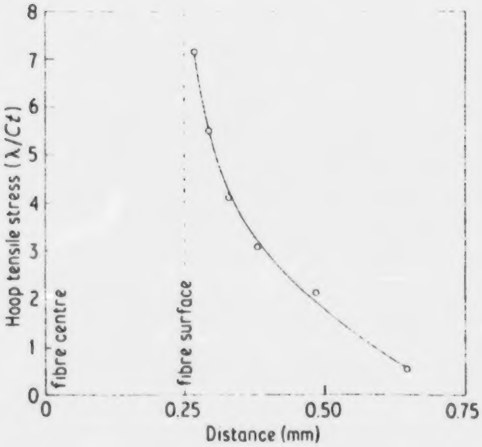
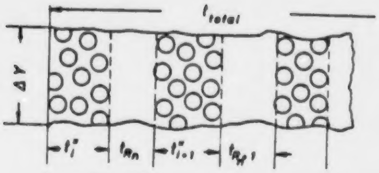


FIG. 34: PLOT OF THE HOOP TENSILE STRESS AGAINST RADIAL DISTANCE FROM THE FIBRE[75]

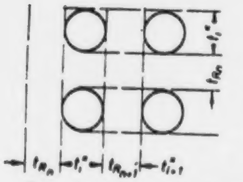


$t_{total} = \sum_i t_i + \sum_n t_{R_n}$ t_{total} = wall thickness

$\delta_R = \dfrac{\sum_n t_{R_n}}{t_{total}}$ (total share of resin parts)

$\bar{\psi} = \psi^*(1 - \delta_R)$ $\bar{\psi}$ = glass volume fraction

Fig .1 Inhomogeneous fiber distribution in a (statistically) representative volume element



t_{total}; $\bar{\psi}$

t_{R_n} = const.

t_i^* = const.

$\delta_R = \dfrac{\sum_n t_{R_n}}{t_{total}} = 1 - \sqrt{\bar{\psi}}$

$\psi^* = \sqrt{\bar{\psi}}$

Fig .2 Layer with homogeneous fiber distribution

FIG. 35: SCHEMATICAL REPRESENTATION OF RESIN AND FIBRE DISTRIBUTION[216]

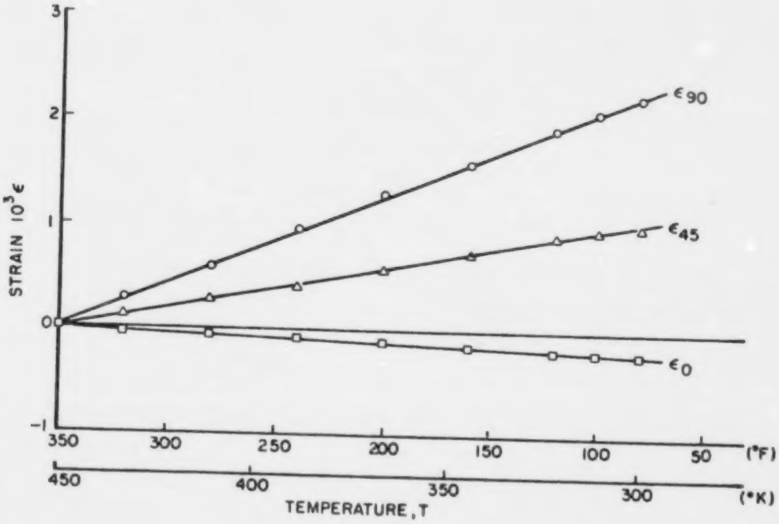
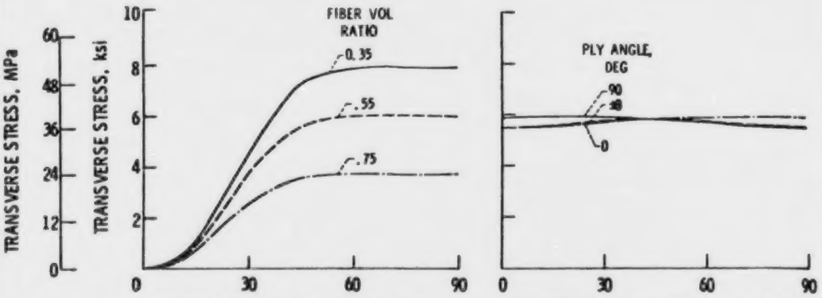
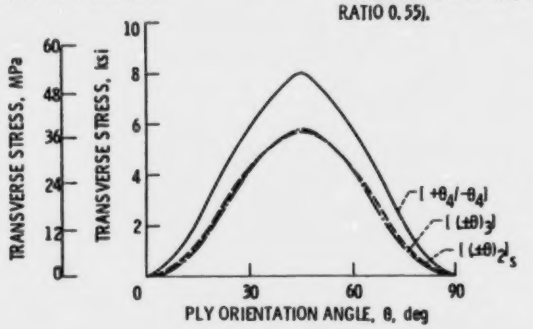


FIG. 36: RESIDUAL STRAINS IN 0-DEG PLIES OF $[0_2/\pm45]_s$ BORON-EPOXY SPECIMEN[78]



(a) STRESS IN THE +θ PLY OF $[0/\pm\theta]_s$ ANGLEPLIED LAMINATE.

(b) STRESSES IN THE PLIES OF $[0/\pm\theta/90]_s$ ANGLEPLIED LAMINATE (FIBER VOLUME RATIO 0.55).



(c) STRESSES IN THE OUTER PLY (FIBER VOLUME RATIO, 0.55).

FIG. 37: PLY RESIDUAL TRANSVERSE STRESS FOR GRAPHITE THORNEL-50/EPOXY COMPOSITES. TEMPERATURE DIFFERENCE, 167 K (300°F)[53]

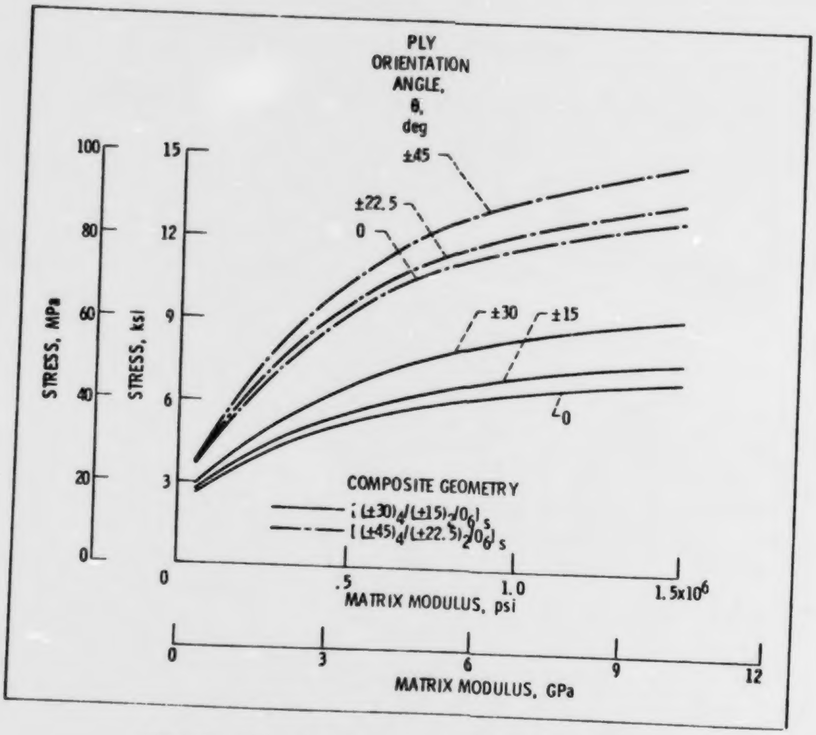


FIG. 38: EFFECT OF MATRIX MODULUS ON PLY RESIDUAL TRANSVERSE STRESS. MODMOR-I/POLYIMIDE COMPOSITES; ZERO VOID CONTENT FIBER VOLUME RATIO, 0.50; TEMPERATURE DIFFERENCE, 333 K (600°F)[53]

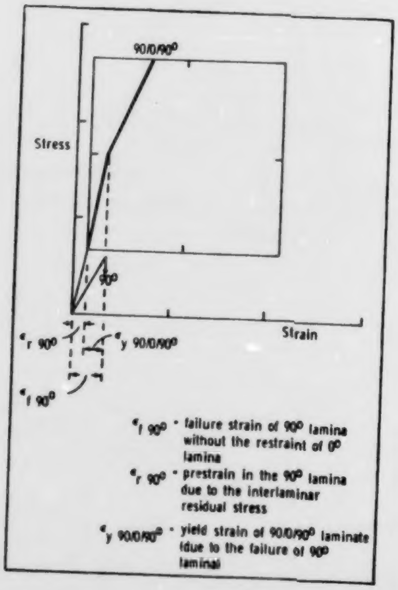

90/0/90°
Stress
90°
Strain
$\epsilon_{r\,90°}$
$\epsilon_{y\,90/0/90°}$
$\epsilon_{f\,90°}$
$\epsilon_{f\,90°}$ - failure strain of 90° lamina without the restraint of 0° lamina
$\epsilon_{r\,90°}$ - prestrain in the 90° lamina due to the interlaminar residual stress
$\epsilon_{y\,90/0/90°}$ - yield strain of 90/0/90° laminate (due to the failure of 90° lamina)

FIG. 39: APPROXIMATE RELATIONSHIP BETWEEN THE YIELD STRAIN OF A 90/0/90° LAMINATE AND THE FAILURE STRAIN OF A 90° LAMINA (WITHOUT THE RESTRAINT OF A 0° LAMINA)[188]

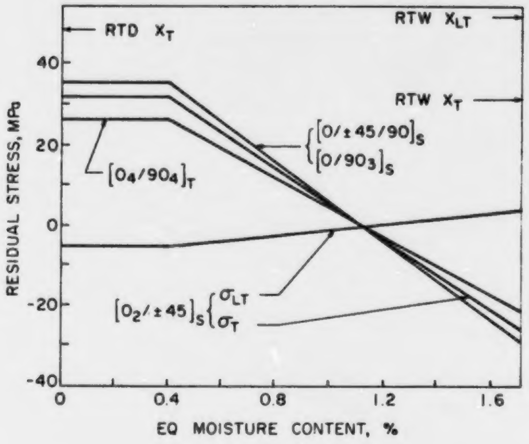


FIG. 40: RESIDUAL STRESSES VERSUS EQUILIBRIUM MOISTURE CONTENT FOR VARIOUS LAMINATES[118]

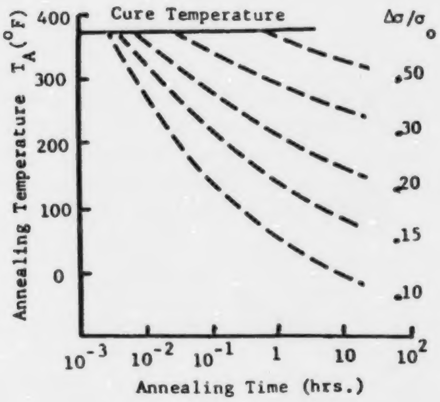


FIG. 41: KINETICS OF 1031 STRESS RELAXATION AS A FUNCTION OF ANNEALING TIME AND TEMPERATURE[73]

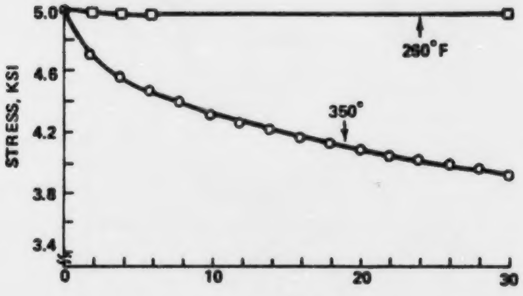


FIG. 42: STRESS RELAXATION OF HERCULES 3501-5A RESIN[128]

APPENDIX A — BIBLIOGRAPHY

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1. Carbon fibre reinforced plastics
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SUMMARY/SOMMAIRE

This report is second in a series of literature reviews in which hygrothermal effects on aerospace composite materials (CM) with polymeric matrixes are examined. This report (Part II) deals primarily with the glass transition temperature and expansion properties due to temperature and moisture. The chapter on residual stresses is also included in this part as these stresses are a direct consequence of the expansion properties. Specific heat, thermal conductivity and emittance are briefly mentioned in Chapter 3.0, while properties connected with moisture absorption have been reviewed extensively in Part I of the series.

Other reports in this series deal with the following topics:

Part I:	Thermal and Moisture Diffusion
Part III:	Mechanical Properties 1
Part IV:	Mechanical Properties 2
Part V:	Composite Structures and Joints
Part VI:	Numerical and Analytical Solutions
Part VII:	Summary of Conclusions and Recommendations

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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART III: MECHANICAL PROPERTIES 1 STATIC TESTS

by

J. P. Komorowski
National Aeronautical Establishment

OTTAWA
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EFFETS HYGROTHERMIQUES DANS LES COMPOSITES À RENFORT DE FIBRE CONTINU

PARTIE III: PROPRIÉTÉS MÉCANIQUES 1 — ESSAIS STATIQUES

by/par

J.P. Komorowski

National Aeronautical Establishment

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W. Wallace, Head/Chef
Structures and Materials Laboratory/
Laboratoire des structures et materiaux

G.M. Lindberg
Director/Directeur

SUMMARY

This is Part III of a series of literature reviews on hygrothermal effects on polymer matrix composite materials. It contains a review of papers on mechanical properties as measured in static tests and includes the effect of impact damage interaction with environmental conditions.

The other parts of the review are:

Part I: Moisture and Thermal Diffusion
Part II: Physical Properties
Part IV: Mechanical Properties 2
Part V: Composite Structures and Joints
Part VI: Numerical and Analytical Solutions
Part VII: Summary of Conclusions and Recommendations

A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.

RÉSUMÉ

Voici la partie III d'une série d'études documentaires traitant des effets hygrothermiques sur les matériaux composites à matrice de polymère. Elle contient une analyse des documents portant sur les propriétés mécaniques telles que mesurées lors d'essais statiques; les effets des conditions environnementales sur les dommanges par chocs y sont également étudiés.

Les autres parties de cette série sont les suivantes:

Partie I: Diffusion de l'humidité et de la chaleur
Partie II: Propriétés physiques
Partie IV: Propriétés mécaniques 2
Partie V: Structures et joints composites
Partie VI: Solution numériques et analytiques
Partie VII: Résumé des conclusions et recommandations

Une liste complète des références est incluse en annexe et les nombres entre parenthèses dans le texte se rapportent à cette liste.

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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART III: MECHANICAL PROPERTIES 1 – STATIC TESTS

1.0 INTRODUCTION

During the past ten years numerous papers and reports have been published on the effects of moisture and temperature on polymeric matrix composite materials. The importance of environmental testing is recognized to the point where design allowables for existing materials or any new material cannot be developed without it[99, 185, 257].

In this part of the review (Part III) results of mechanical tests on composite materials are reported. The tests of interest were tension, compression, torsion, in-plane shear, beam tests for interlaminar shear and flexure tests. Numerous types of specimen design were used in these tests and the properties reported are usually strength, modulus and notch or impact damage sensitivity. In order to assess the amount of degradation of mechanical properties which might be expected to occur due to temperature and humidity history, samples were often exposed to various conditions prior to and during the test.

Environmental effects on composite materials properties as measured in fatigue and creep tests will be reported in Part IV of the review.

Bolt bearing capabilities of composites in a varying environment will be reviewed in Part V.

2.0 MATERIALS UNDER CONSIDERATION

The effects of moisture and temperature on a multitude of composite materials have been reported. Some of the materials studied are very exotic and have never reached commercial use, while others have been commercially available for several years. The results presented below should be treated as a rough guide to the effects on various types of fiber reinforced resins. The performance of composite materials may vary substantially depending on the molecular structure and processing history of the particular resin used, and this is illustrated in Figure 1 with reference to different composites fabricated from Thornel Fabric 133. Terms like 'epoxy' or 'polyimide' resin are very loose descriptions of the matrix and cover a wide variety of chemical structures.

Clements and Lee[66] used unidirectional T300/5208 samples to study the effects of quality control variables such as batch variation, postcure and specimen quality under extreme moisture conditions. They found that in the fiber direction, differences between batches produced greater differences in strength and modulus than did the high level of absorped moisture. However Chen and Hunter (Boeing)[59] pointed out that mechanical property tests are not adequate for quality assurance purposes. Slight differences in chemical content and cure quality may result in materials with equivalent short term mechanical properties, but having different rates of degradation due to the environment. For this reason Boeing is committed to very accurate material characterization methods supported by mechanical property tests.

The need for precise characterization of the resin has been recognized and a good example is the work of Lamothe, Halpin and Neal[185], who were engaged in developing design allowables for glass/epoxy (S2-449/SP-250) for use in MILHDBK 174. Allowables include HPLC (High Pressure Liquid Chromatography) for checking component weight percent of SP-250 and FTIR (Fourier Transform Infrared Spectroscopy) for cure quality control. However, specimens used in most references under review have not been prepared under strict controls and therefore results should be treated as indications of the environmental stability of a given material.

3.0 TEST METHODS AND SPECIMEN DESIGN

Tension and beam tests are most frequently used. Tensile specimens are relatively easy to produce and the loading method is simple. Various specimen shapes have been used, and these are usually based on ASTM standards (i.e. ASTM 03039). These tests are primarily used to determine unidirectional tensile strength but off axis tensile specimens (10° or ±45°) are also employed to measure changes in in-plane shear strength and modulus. Less common are notched shear specimens (see Fig. 2) also loaded in tension. Chapman [58] has described tension testing at elevated and cryogenic temperatures for unidirectional and ±45° specimens of graphite/polyimide. Special attention was paid to the application of strain gauges.

The beam tests are of two types: flexure and short beam shear, they can both be either three or four point bend. The differences are in the ratio of span to depth. These are the easiest tests to prepare and perform under varying conditions, as tabs are not needed and the fixtures developed for room temperature can be used.

Torsion tests for shear were used by Adsit[5] with torsion tubes, while Philips, Scott, Buckley[233] and Hancox[122] used solid rod specimens.

Garcia and McWhithey[104] did both experiment and finite element analysis of rail shear test specimens made for graphite/polyimide and concluded that it is a good method for measuring the in-plane shear modulus but not strength.

For compression testing, several methods have been used in conjunction with environmental conditioning and this seems to be the most demanding of all tests. Good references for these types of test are Adsit[5], Grimes[115], Camarda[47] and Shuart[266].

All of these tests provide information on mechanical properties which can be grouped into two categories -- fiber or matrix dominated properties. Whether the test provides the information on either of these property categories depends on both specimen loading and geometry (lay-up) i.e. a unidirectional specimen loaded in transverse direction to the fibers provides information on the matrix dominated properties. Interpretation is not straight forward as the specimen may fail in a mode where fiber splitting is dominant[11]. Generally, it can be said that 0° tension and flexure tests provide information on fiber dominated properties while other tests are matrix (or interface) dominated. This is important as most fibers demonstrate good resistance to temperature and moisture changes (Kevlar and other organic fibers are exceptions to this rule[11]) and it is usually the matrix or interface which shows degradation.

One of the great advantages of composite materials is the capability of changing laminate properties through various lay-up arrangements. The number of possible combinations, especially when hybridization is included, is almost infinite and it is not feasible to test all of them fully. Most frequently unidirectional and crossplied samples are tested and results from these tests are used with lamination theory to compute the effects of different lay ups. As residual and swelling strains significantly alter the overall performance of a laminate and are not easily accountable for in calculations, a limited number of tests must be conducted on the most frequently used lay-ups. The results of these tests are compared to theoretical results (lamination theory). Residual and swelling strains will be discussed in Part II of the series.

4.0 NOTCHED AND IMPACTED SPECIMENS

For practical applications the notch sensitivity of a material is an important factor. It also gives an indication of the damage and defect tolerance of the material. Low energy impact resulting in damage on the threshold of detectability has limited the design allowable strains to about 4.10^3 μmm/mm in present day graphite/epoxies[99]. It has been shown that such damage will not propagate if post-impact strain values are kept below this limit.

Interactions between the notch, internal damage and the environment are other important factors requiring consideration. Wilkins[316], Porter[235], Bailie et al[27], Lauraitis and Sandorff[186] all studied these type of interactions. Their results were similar despite the use of different materials, lay-ups and tests. Wilkins studied T300/5208 samples containing holes. Specimens with different lay-ups were saturated with water (1.5% weight gain) at 82°C and 98% RH. Tensile tests were carried out at room temperature and at 93°C on dry and wet specimens. Porter used T300/934 samples with various holes, notches and defects and three lay-up configurations. Conditioning was similar to that used by Wilkins except that instead of exposure at 98% RH immersion in water was utilized. Tensile testing was carried out at room temperature and 149°C with thermal spikes applied to reach testing temperature in 60 seconds and loading in 180 seconds after beginning of heat up. Bailie et al studied the influence of holes on cloth and tape laminates (HMF 330C/34 and AS/3501-6). Specimens were loaded in compression and their strength was determined at temperatures up to 157°C. Lauraitis and Sandorff also loaded their specimens in compression. They studied the interaction of moisture, low temperature and low level impact in AS/3501-6 material conditioned up to 1.29% and 1.75% weight gain of moisture.

The results of these tests led to the conclusion that notched or impacted material strength is only slightly dependent on lay-up and that there is no clear difference in notch or defect sensitivity as testing temperature or moisture content changes. This is probably because at higher temperatures notch stress concentration is reduced as well as the strength of the material. These results are significant and were best summed up by Porter[235] who concluded that environmental conditions generally have the greatest effect in the noncritical design condition of no defect. It has to be borne in mind, however, that only static loads were used in these tests. The same may not be true in fatigue tests.

5.0 TEST PROCEDURES -- TEMPERATURE AND HUMIDITY PROBLEMS

The test conditions which have been employed are: 1) constant temperature (high or low), 2) temperature cycling including thermal spikes, 3) constant humdity including immersion, 4) combinations of varying temperatures and humidities and 5) natural weathering on racks. Very few specimens have been made from components that have been in actual service for a number of years. This is partly due to the fact that data is often not available on the initial mechanical properties of many of the earlier composite materials employed.

Since advanced composite materials are exposed to a wide range of temperatures during their service (see Chapter 2 of Part I on environmental conditions), testing of conditioned specimens has to be carried out under similar ranges of temperature, and should include both the dry and wet states. Very often, in connection with determining mechanical properties, the question of reversability of property changes due to moisture is raised. Upon drying the property is usually restored or at least partially so. This is important in understanding the mechanisms of degradation, but it is irrelevant if one is only interested in developing design allowables, since, these should take into account the worst possible case.

Testing of composites at room temperature is difficult and standards have not been widely accepted for all types of tests. This applied specifically to shear and compression tests. The problems are even greater for tests conducted under hot/wet conditions. There is a paucity of data that might shed light on these problems. Some researchers have reported on the problems of conducting mechanical property tests in hot/wet conditions. Shen and Springer[261] studied the effects of temperature and moisture on the tensile strength of composites. They have used a computer program to estimate the thickness of the layer affected by three minutes of drying at different temperatures and the moisture loss of specimen in these conditions. Some of the results of these computations for T300/1034 system are presented in Figure 3. While loss of some moisture and consequently changes in moisture distribution during testing has to be expected, losses should be minimized. Frequently the time at temperature prior to testing is not reported and "soak times at temperature" of up to 30 minutes prior to testing are not uncommon.

Whitney and Husman[314] described the use of a flexure test for determining the environmental behavior of composites. Notably the authors recognized the need to control the weight loss. As 90 seconds were needed to bring their 8-ply T300/5208 and AS13501-5 specimens to temperature equilibrium, they calculated corresponding weight loss at the highest test temperature of interest. The authors assumed that the coefficient of diffusion is constant with temperature and calculated soak times at lower temperatures required to obtain similar weight losses.

6.0 FAILURE MODES

The ultimate goal of materials testing is development of data sets for designers (design allowables). For CM it became obvious that this data is closely related to the definition of failure. As failure mode is affected by the testing method and conditions in which it is carried out it is very important how the failure is defined. Tests carried out on CM structures have to be related to the design allowable data and this can only be done if the same failure has been induced in the structure as in the earlier coupon and element test.

Whitney and Husman[314] demonstrated that flexure testing provides a simple means for determining environmental conditions which induce significant changes in mechanical behavior related to flexure-stress conditions. Their results showed that moisture and temperature induce a change in failure mode from filament dominated to matrix dominated.

If the results obtained in hot/wet conditions are to be meaningful and provide a basis for quantitative assessment of the property loss relative to dry room temperature conditions, tests should be designed in such a way as to induce the same failure mode for all conditions. Crossman and Mauri[69] addressed this problem in connection with a interfacial shear strength hygrothermal degradation evaluation. Depending on the span to depth ratio used and on environmental conditions a bend test provides information on either the flexural strength or on the interlaminar shear strength. Crossman and Mauri demonstrated that the four point bend test gives wider than three point bend test range of span to depth ratios for which an interlaminar shear failure can be induced. Once the proper ratio is chosen for dry room temperature conditions failure should always be through interlaminar shear. This may not be true for flexure where a transition to shear failure may occur if span to depth ratios are not sufficiently high.

Changes in the failure mode with higher moisture content were also reported[267] for compression tests. In spite of great care in specimen preparation and test method design, tab failures in tension and compression tests are reported, i.e.[115]. Adsit[5] has discussed test methods and procedures that have been used at elevated temperatures for graphite/epoxy and polyimide composites. Tests include flexure, tension, compression and shear and test temperatures of up to 370°C are reported. This work should be of interest to anyone involved in environmental testing of CM.

7.0 FRACTURE SURFACE ANALYSIS

Miller and Wingert[217] demonstrated that despite the fact that fracture process is complicated, fracture surface analysis does yield information showing fundamental differences between resin systems and influences of environment. For this purpose scanning electron micrographs (SEM's) of gold plated specimens are most suitable, while optical microscopy on polished surfaces may provide additional information.

8.0 FRACTURE MECHANICS AND ENVIRONMENTAL EFFECTS ON PROPERTIES

Success in the application of fracture mechanics to composites has been limited since the models developed for metals cannot be directly applied. However, Beaumont and Harris[31] measured the work of fracture required to fracture unidirectional carbon fiber reinforced epoxies. They tried to establish the nature of the major energy absorbing fracture processes and to isolate mechanisms which influence crack motion. Some samples were exposed to water at 23°C and steam (100°C) up to two weeks prior to testing.

A similar approach was adopted by Kaelble et al in series of articles[157, 158, 159, 156]. They concentrated on developing theory and analysis of fracture energy and have studied the influence of degradation of interfacial bond strength upon fracture energy. Permanent degradation of bond strength was reported with a simultaneous increase in fracture energy.

Mandell[205] studied the effects of moisture on crack propagation in fiberglass laminates. While water immersion caused reduced static and fatigue tensile strength and consequently increased crack extension rates under cycling loading, it greatly reduced crack extension under static loading. It was shown by the author that this was due to moisture increasing the intraply delamination region in the damage zone thus reducing the local fiber stresses. For cyclic loading, this region is large even in dry conditions and reduction of material strength plays a dominant role.

9.0 EFFECT OF HUMIDITY AND TEMPERATURE ON VARIOUS MATERIALS

9.1 Graphite and Boron Composites for Service Up to 200°C

Degradation due to exposure at constant temperature for extended periods of time has been studied by Haskins, Kerr and Stein[166, 127]. After 10,000 hours at 121°C samples of AS/3501 in $[0]_6$ and $[0/\pm45]_{S6}$ lay-up which were tested at 177°C showed no degradation of tensile strength. Similar samples aged at 177°C after 1000 hours began to degrade, and after 10,000 hours demonstrated 20% and 57% decrease in strength at 177°C when compared to unaged samples. The greater amount of degradation was observed for a $[0/\pm45]_{S6}$ lay-up.

Kong et al[174] observed the effect of quenching from above the glass transition temperature (Tg) and subsequent Tg quenching for up to 10^5 min. on T300/5208 $(\pm45)_{4S}$ laminates. Decrease of ultimate tensile strength (32%), strain to failure (93%), and toughness (68%) after 10^5 min. were interpreted as physical aging effects and were explained in terms of decreases in free volume and the tendency to move towards thermodynamic equilibrium in the glassy state.

Thermal cycling effects were reported by Camahort, Rennhack and Coons[46]. Samples were cycled 25 times between −196°C (liquid nitrogen) and 100°C (boiling water). Materials used were HMF 330C/934 fabric and HMS/934, HMS/3501 and HMS/759 unidirectional tape graphite/epoxies. All these materials were specified as 177°C cure but in order to reduce residual stresses were cured at 135°C. Some samples were made from HMS/339 which is a 121°C cure material. Microcracking was severe in all but the 121°C cured materials. However, there was no loss in RT tensile properties with some improvement in transverse tensile strength. These results are not surprising as TTT diagrams for thermosets indicate that the materials were not fully cured.

Mazzio and Mehan[209] thermally cycled HTS surface treated fibers in Epon 828 (manufactured by Shell) cured with hexahydropthalic anhydride (HHPA) and benzyldimethylamine (BOHA) (100:78:1). The cycle temperatures were between −53°C and 149°C of which 250 and 500 were slow cycles and 500 were fast cycles (see Fig. 4). Results are presented in Figure 5. Mazzio and Mehan prepared some samples with a lower volume fraction of fibers and studied residual stress effects optically. Fibers after cure were in compression for low v_f, and occasionally fiber buckling was observed. During cycling some of these stresses may relieve themselves by breaking molecular bonds and as a result mechanical properties will change. A decrease in compression strength seemed to confirm this but the authors appear to have contradicted themselves by saying that interlaminar shear strength increased through improved interfacial action. No effect of cycling rate was observed.

Loos and Springer[198] studied the effect of thermal spiking on Graphite-Epoxy T300/1034 composites. The material properties studied included moisture absorption, tensile strength and buckling modulus. No effect was observed in the properties that were studied. Loos and Springer compiled results of other researchers (see Table 1) and concluded that the effect of spiking depends on the particular material system being studied. In most cases, the effect is small with fiber dominated properties especially insensitive. See also[200, 201].

In[267] Shyprykevich and Wolter determined compressive strength as a function of moisture content. AS/3501-5A was used in two different lay-ups and testing was performed at room and elevated (127°C) temperatures. At higher humidities (>1.2% wt gain) thermal spiking clearly degraded the material, and this was related to glass transition temperature (Tg) exceedences. Thermal spiking caused higher moisture absorption which lowered the compressive strength and also changed the failure mode.

Browning, Husman and Whitney[41] tested AS/3501-5 in various lay-ups after near equilibrium moisture gain at 71°C (75% and 95% RH). Test temperatures were RT, 93°C, 121°C and 149°C. Typical results are presented in Figure 6. Browning et al suggested three possible mechanisms of degradation: 1) matrix plasticization associated with Tg, 2) degradation due to residual and swelling stresses and 3) possible degradation of the interface. Tests on neat 3501-5 led Browning et al to conclude that room temperature properties are degraded due to matrix crazing.

Shen and Springer[260, 261] studied the effect of moisture and temperature on tensile strength and buckling moduli of T300/1034 in various lay-ups. Different conditioning temperatures and humidities were employed. The moisture distributions and losses due to testing in high temperature were calculated. Typical results are presented in Figure 7 and were compared to previously reported data (see Table 2 and 3). Results are consistent and indicate that:

a) Temperature and moisture had little effect on fiber dominated longitudinal strength below 100°C and slightly decreased strength at 180°C. Elastic moduli was not affected regardless of moisture and temperature up to 180°C.

b) Transverse properties were severely degraded with increase in temperature and moisture and both the modulus and strength maybe degraded by as much as 90% of their original values.

c) Distribution of moisture in 0° and quasi-isotropic laminates did not seem to affect the results. For 90° specimens the moisture distribution may have influenced the absolute value of ultimate strength and modulus but it was unlikely to have affected the trend in the data.

Bohlmann and Derby[38] used sandwich beam specimens to test T300/339 fabric (HMF-330B) in the warp direction. Specimens were conditioned at 82°C and 70% RH and tested at both room temperature and 177°C.

Grimes[115] made extensive studies of AS/3501-6 material in compression. As different lay-ups were tested, various loading fixtures were utilized. Testing was conducted at room (22.8°C) and elevated (103°C) temperatures, with specimens in dry (0 - 0.4% wt gain) and wet (1.1 ± 0.2% wt gain) strabes. Typical results are shown in Figure 8.

Lauraitis and Sandorff[187] studied the effect of environment on the compressive strength of T300/5208 and AS/3501-5A in various lay-ups. Specimens were soaked up to equilibrium wt gain at 82°C - 90% RH and tested in -54°C, 22°C, 93°C and 135°C (above "wet" Tg). Behavior of graphite/epoxy laminates was divided into three regimes:

1) Long column range — elastic stability.

2) Very short column — specimens fully supported failure by crushing, delamination (compression ultimate).

3) Intermediate short column — inelastic failures resulting in rupture.

It was found that in regime 1) the behavior of the laminate was independent of temperature and humidity while in 2) and 3) the environment affected results depending upon material and lay-up.

Ekvall and Griffin[99] performed over 2000 coupon tests to develop design allowables for T300/5208 tape and fabric laminates with a 3/16-inch hole, at −54°C, room temperature dry and 82°C wet (1% weight gain condition). Data were analyzed statistically to determine the best fit propability distribution and the results at the lower tail of the test data were compared to the minimum values obtained from different regression equations. An overall best fit equation (in this case normal distribution) provided good agreement near the lower tail of the distribution. Final design allowables for worst environmental conditions (in this project), 3/16-inch diameter notch and nonvisible impact damage were calculated (Table 4) and included a statistical reduction factor (B allowable factor). This methodology can be used for other composite materials.

Several authors published data from shear tests conducted on pre-conditioned samples. Philips, Scott and Buckley[233] used rod specimens prepared from graphite, glass and Kevlar 49 fibers in HY750 epoxy resin. Results are shown in Table 5 and Figure 9. Crossman, Mauri and Warren[69] used four point beam tests to study interlaminar shear properties of T300/5208 and T300/5209 specimens. Conditioning was 55% RH or 85% RH at 70°C with some samples dried. Testing was conducted at 20°C, 70°C and 93°C. Several specimens were subjected to temperature cycles varying from −56°C to 70°C or 93°C. The results led the authors to conclude that neither testing temperature nor conditioning significantly degraded T300/5208 dried samples. However, T300/5209 showed significant loss of interlaminar shear strength (ILSS) after humid hot conditioning and this seemed to be related to the exceedence of Tg (5209 is a 121°C cure material). For low moisture gain T300/5209 does not seem to be affected.

Halloff[121] tested short beam shear (SBS) strength of T300/5208, HT-S/3501 and Fibredux 914C. Samples were subjected to different and sometimes severe temperature and humidity treatments. However, Halloff concluded that the chemical composition of the polymer system most significantly influenced the effects of heat treatments and moisture absorptions of composite materials.

Lifshitz[193] measured longitudinal, transverse and axial shear properties of T300/5208 laminates. The test program included three strain rates, three temperatures and three levels of moisture. The matrix properties were influenced by all those parameters with axial shear exhibiting the most variability (Fig. 10). Poisson's ratio v_{12} did not change in these tests, however, the value was slightly less than usual.

Augl in[23] presented the effects of moisture and test temperature on T300/5208 as demonstrated in beam tests. Figure 11 demonstrates the usefulness of three dimensional (3D) graphs in reporting degradation of strength due to moisture and temperature.

All above reported tests were done on samples subjected to arbitrarily chosen conditions. It is not known whether materials in real service, subjected to atmospheric conditions, would be degraded to the same extent. To gain knowledge on the natural weathering of composites, several materials have been exposed on racks in loaded and unloaded state and some tests were carried out on specimens recovered from composite structures after extended period of service.

Trabocco and Stander[291] used two exposure sites (Panama Canal and Warminster, PA) to represent tropical and temperate climates. Specimens were either painted or unpainted and loaded in flexure with strain at 4000 μmm/mm. Tests were carried out at room temperature and at 177°C (Fig. 12). The materials that were used in this work have been replaced by those currently used in aerospace industry (AS/3501-6, T300/5208 and AS/3004). The results for these new materials were reported by Vadala and Trabocco[294]. Two aircraft carriers operating in the Pacific were included as exposure sites. Tests were carried out at room temperature, 82.2°C and 121.1°C with materials originally intended for 177°C service limited to 121.1°C. Tension, flexure, compression (IITRI compression fixture) and SBS tests were carried out. Weight gains were monitored but specimens were kept in sealed bags prior to testing. However, sealed bags do not prevent changes in moisture distribution. Typical test results are shown in Table 6 and Figure 13. In general Panama exposure was slightly more severe than at Warminster. The rate of decrease of strength varies for each material system with AS/3004 (graphite/polysulfone) showing better resistance to environment than epoxies.

In[57] Chapman, Hoffman and Hodges and in[88] Dexter and Chapman presented some results obtained by NASA for commercial aircraft composite laminates. Composites were exposed at ground exposure sites around the world and in actual service. No significant deterioration was observed owing mainly to mild testing conditions. Information on specimen lay-up and of failure modes was not given. See also[29, 199, 30].

Gibbins and Hoffman in[110] report on a long term program by Boeing aimed at developing accelerated aging methods for composites used in commercial aircraft. Various graphite fiber and one Kevlar fiber in epoxy materials were tested after exposure to actual flight conditions, on ground and in accelerated laboratory chambers conditions. In[110] results obtained up to now are presented (2 years of the projected 10 years of total program duration).

Coggeshall in[67] reported on the Boeing 737 graphite composite spoiler flight service evaluation. This is the seventh annual report on a program involving 114 spoilers installed on 27 aircraft operating around the world. Various types of materials were used in the spoilers and samples are also exposed on the ground. Periodically some spoilers and ground exposed samples are mechanically tested. No significant deterioration of properties has been reported up to now.

9.2 Glass Composites for Service Up to 200°C

Glass composites have been available since the 1950's and studies on environmental degradation of these materials were initiated in the 1960's. The degrading effect of moisture and temperature on matrix dominated properties is similar to the graphite reinforced composites which basically depends on type of resin used.

It has been found that glass fibers are degraded by water due to a reaction between alkali ions in the glass and water which in turn affects the siloxan bond (the structural backbone of glass), forming an alkaline medium on the surface of the glass. If uncoated glass is used in a resin matrix the rapid migration of water along the fiber interface is observed. This is paralleled by degradation of the interface and tensile strength of the fiber. Present day glass fibers are used in strongly-bonded polymeric coatings and a considerable amount of research has concentrated on the reinforcement mechanism of glass fiber reinforced plastics. Ishida and Koenig[142] reviewed literature on this subject with an emphasis on the microscopic aspects (molecular structures of glass/matrix interfaces). The glass/matrix interface was studied as a three-phase system and includes a) glass/coupling agent interface, b) coupling agent and c) coupling agent/matrix resin interface. Each of these phases was studied individually using modern spectrometers.

Antoon and Koenig[14] used Fourier-transform infrared (FTIR) spectroscopy to identify irreversible chemical effects of moisture on highly cross-linked anhydride-cured epoxy resin. The effects of high tensile stresses and presence of filler were also investigated. It was concluded that hydrolitic attack of water on the ester linkages was accelerated in the alkaline media, enhanced by the presence of inorganic filler (glass fiber) and was a mechanically activated process (external or residual stresses). Further studies of the interface were reported by Koenig in[172]. Spectroscopic studies should be correlated with mechanical property changes and are indispensible in understanding degradation mechanisms. A good example of such a study was work by Ishai[141] where samples of glass fabric/epoxy were subjected to several absorption-desorption cycles and their wrap and fill strength was tested in tension. Infrared spectroscopy was used to analyze the water during immersion and distinct traces of silica compounds were found. Ishai pointed out that caution is called for in using elevated temperatures for accelerated testing, since correlation between short-term effects and their long-term counterparts at low temperatures is not clear.

Scola[255] studied shear and flexure properties of three types of S-glass/epoxy composites and their resins after various temperature and humidity conditions. The typical results are presented in Figure 14. One interesting observation was that the principal cause of composite shear strength loss due to water is weakening or debonding of the resin-fiber interface, however, the strength and modulus changes of the resin appear to be insignificant in the degradation process. Some degrading influence of the resin was found by Scola in a further study[254].

Nicholas and Ashbee[226] studied the effect of freezing or boiling of phase-separated water, and concluded that: 1) non-spherical water-filled cavities, specifically disc shaped cavities present at water soluble inclusions propagate as cracks during the volume expansion associated with the water-to-ice transition, 2) generation of the osmotic pressure is also responsible for the observed failure of phase-separated water to boil during high temperature excursions.

Docks and Buck[92] subjected several glass fiber/resin composites to thermal cycling and grouped the resins according to their susceptibility to degradtion in these conditions. Some vinyl or modified vinyl resins seemed to perform best.

Rao et al[238] measured the effect of moisture and glass contents on the Poisson's ratio of glass fiber reinforced plates. Laser interferometry was used to measure Poisson's ratio and the results are in Figure 15.

Lamothe, Halpin and Neal[185] developed design allowables for glass/epoxy (S2-449/SP-250). After the test specimens were subjected to different preconditioning cycles, tension, compression and SBS tests were carried out, Table 7.

Lubin and Donohue[199] presented unique and encouraging data from tests carried out on samples of glass fiber composites cut from aircraft structures which were in service for up to 19 years and the results were compared with data obtained at the time the structures were manufactured. Some of the results are in Tables 8 and 9. Most of specimens showed excellent strength retention, but, only a few of these parts were exposed to temperatures over 82°C which may be the reason for the low degradation.

9.3 Aramid (Kevlar) Fiber Reinforced Composites

Despite the growing amount of Kevlar composites being used in aerospace and other industries relatively little work has been published on hydrothermal effects on these composites. Kevlar fibers are organic and, in contrast to carbon or glass fibers, they absorb moisture.

Wu[322] studied strength degradation in filament wound Kevlar 49/XD7818, Jeffamine T403 epoxy. Samples tested in longitudinal and transverse tension, and in longitudinal compression were the flat coupon type. Transverse compression and shear was measured on tube specimens while biaxial tests were performed on pressurized tube specimens. All specimens were dried in dessicant and then subjected to one of the following conditions: 52% RH at 23°C, water at 23°C or water at 100°C until moisture equilibrium was reached. The typical effect of moisture on ultimate strength is shown in Figure 16, which indicates that substantial degradation occurs for room temperature water immersion. (However, in service this is not likely to occur as much lower wt gains are observed.)

Wu concluded that due to large differences between tensile and compressive strengths and different degrees of degradation in the fiber-controlled vs matrix controlled strengths which caused the failure surface to shift and deform, a polynomial third order tensor strength criterion would be more suitable. However, this required four additional biaxial experiments to determine coefficients for the polynomial.

Allred[10] studied the effects of temperature and moisture content on flexural response of two Kevlar 49 fabric (181)/epoxy laminates (5208 and Ferro CE-9000 both 190°C cure) in two lay-ups [0/90] and [±45, 0/90]. The testing temperature range was from −55°C to 150°C. Both materials exhibited fairly similar properties. For both lay-ups the results indicated that Kevlar 49/epoxies have temperature dependent mechanical properties over the range investigated, and that moisture increased temperature sensitivity. Load vs deflection for [0/90] CE-9000 is shown in Figure 17.

In flexure tests at 150°C and near the moisture saturation content (5% wt), a loss in strength of 60-70% and effective stiffness loss of 40% was recorded. At 21°C the saturated specimens were 35-40% weaker than dry material. Specimens having a [±45, 0/90] lay-up seemed to be slightly less sensitive to moisture. Failure modes which were distinctively different then for other composites were reported, i.e. compression buckling of filaments of [0/90] or tensile delamination for the [±45, 0/90] lay-up.

For the [±45, 0/90] lay-up the effects of voids, long term moisture exposure, freeze-thaw cycling and reversibility of moisture exposure on drying was also investigated. All these conditions were found to degrade laminate strength and degradation due to moisture exposure was found to be irreversible (dried specimens had the same strength as specimens saturated with moisture at room temperature).

Deteresa et al[86] used single filament techniques to establish the critical length of Kevlar 49 fiber in polyaramid (Nylon 6) film. Tests were performed at room temperature. It is surprising that soaking in water seemed to shorten the critical length apparently strenthening the bond between the fiber and matrix, but further studies are required to clarify this point.

Humphrey et al[136] subjected Kevlar 49, filament wound composites with various resins to 71°C and 95% RH. After 21 days of exposure specimens tested in short beam shear demonstrated reduction in strength from 29% to 46% depending on type of resin.

Kevlar 49/Fiberite 934 epoxy composite, in transverse direction was tested by Allred and Roylance[11]. At 25°C moisture saturation ($>$ 5% wt gain) caused a 35% decrease in ultimate strength and 25% decrease in elongation. Stiffness was found to be less sensitive with a 14% decrease from the dry room temperature value. Microscopy of the fracture surfaces revealed that property reductions were accompanied by a change in failure mode from an interface dominated one in dry condition to a filament splitting mode in the moisture saturation condition.

In 1973, Lockheed under a contract from NASA manufactured and installed Kevlar 49 fairing panels in three L1011 wide body transports. Concurrently, NASA runs ground-based exposure tests on Kevlar 49 coupons. Reference[276] is the eighth annual flight service evaluation report which includes the ground-based coupon test results. The Kevlar 49 fairings continue to perform satisfactorily and no mjaor damage or defects have been observed after eight years of service. However, these components are lightly loaded and so far no tests have been performed to assess the amount of degradation of properties or to measure the moisture content. Tests were carried out only on the ground exposed samples. It was found that moisture contents stabilized after five years at slightly over 2% wt gain. No degradation in flexural strength was found while shear and compressive strengths after five and seven years decreased in the 15% to 20% range.

There are other on-going service evaluation programs, i.e.[30] which should provide more information on degradation of Kevlar composites.

9.4 Composites for Service Above 200°C

An excellent review on synthetic resin matrices for use up to 300°C can be found in a recent book by Delmonte[84]. Recent papers have been reviewed which discuss moisture effects in these materials.

One of earliest works of the effects of water on the properties of a glass/polyimide laminate was reported by DeIasi[83]. After 1200 hours of exposure to 100°C water or 100% RH specimens made of 7781 glass fabric with 1100S finish and Monsanto's Skybond 709 polyimide resin lost 90% of their flexural strength when tested in dessicated state. The calculated value of the activation energy of the degradation process indicates selective hydrolysis of the matrix material followed by a breakdown of the polymer-fiber interface.

Lisagor[194] studied the effect of moisture on short beam shear and compression strengths in HTS2/PMR15 and Celion 6000/PMR15. Specimens were conditioned to saturation at 100% RH and 82°C and tested at -96°C, 21°C and 316°C. Vacuum drying of as processed samples produced improved mechanical properties. Results for "wet" samples should be compared with dessicated samples, i.e. for compression strength compare Figures 18 and 19. Moisture conditioning of graphite/polyimide composites produced moderate to severe reduction in compressive and interlaminar shear properties at 316°C. This degradation appeared to be associated with the lowering of the Tg of the matrix.

Rummler and Clark[250] presented results of thermal aging of HTS/710 for times up to 25,000 hours. Specimens were tested in tension at the aging temperature. The results indicate that HTS/710 material shows degradation after 1000 hours at 288°C and after 10,000 hours it was severely degraded. For lower aging temperature (232°C) no degradation was observed after 25,000 hours. Thus the maximum service temperature for HTS/700 should be reduced to 232°C for long time application in aircraft such as supersonic transports.

Scola[256] investigated the effect of thermal aging and moisture on several composite systems consisting of addition type polyimides PMR-11, PMR-15, P13N in combination with the fiber reinforcements S-glass, Thormel 300, HMS and HTS graphite fibers. PMR-11 and P13N systems proved to be fairly resistant to degradation due to moisture and PMR-11 systems demonstrated good flexural and shear strength retention after 2500 hours in air at 288°C.

Pater[22] showed that PMR resins modified with N-phenylnadimide had superior properties to standard PMR-15. Tests included isothermal exposure at 315°C for up to 1500 hours in air and hygrothermal exposure for 360 hours in 95% RH at 82°C, followed by flexural and shear strength testing at 315°C.

For graphite/polyimides strength degradation is directly associated with fiber thermo-oxidative resistance while stiffness retention seems to be controlled by the thermal stability of the matrix in flexural and shear tests[181].

Serafini and Hanson[259] investigated the effects of thermo-oxidative and hydrothermal exposure on T300/PMR-15 and HTS2/PMR-15 composites. It was very difficult to carry out tests in "wet" condition at 200°-300°C since desorption at these temperatures was very rapid. This cast doubt on the validity of other elevated temperature "wet" results. The case of loaded and soaked material being subjected to temperature spike is not unlikely in acutal service and greater strength reductions for this case can be expected.

10.0 CONCLUSIONS AND RECOMMENDATIONS

Many conclusions can be reached from the preceeding sections and only the most important ones are listed below along with recommendations for further work proposed by the reviewer.

(i) There is a growing need for standards for the testing of composites. The areas that have to be addressed are:

  a) Standard methods are required for material characterization, including chemical content and cure quality.

  b) Specimen and fixture designs are required which take into account the hot/wet conditions used in testing.

  c) Standard methods are required for predicting the realistic moisture contents which can be expected during service.

  d) Standard methods are required for preconditioning the sample before testing in wet conditions.

  e) Standard technique of testing under hot/wet conditions (temperature gradients, humidities, loading rates, etc.).

  f) Greater emphasis should be placed on the statistical analysis of data.

(ii) Environmental conditions generally have the greatest effect on the noncritical design condition of no defect. There is no clear difference in notch or defect sensitivity as testing temperature and moisture content changes.

(iii) Slight differences in chemical content and cure quality may result in materials with equivalent short term mechanical properties but different environmental sensitivities.

(iv) Materials used in most references have not been accurately characterized and therefore the material properties reported may only be treated as indications. This is especially true of environmental sensitivity of composite materials.

(v) Maximum service temperature for some epoxies, formerly advertised as 177°C (350°F) should be lowered generally by 50°C due to hot/wet properties being greatly reduced.

(vi) Fiber dominated properties seem to be little affected by moisture and temperature.

(vii) Results of tests on samples exposed either on the ground or taken from structures that were in actual service for several years, show little degradation due to environmental exposure. However, most of these samples were only lightly loaded.

TABLE 1

SUMMARY OF EXPERIMENTAL DATA ON THE EFFECTS OF THERMAL SPIKES ON GRAPHITE/EPOXY COMPOSITES[198]

Material	Reference	Absorption Behavior	Tensile Strength	Compression Strength	Shear Strength	Flexural Strength	Buckling Modulus	Tensile Modulus	Fatigue
T300/1034	Present work	N	N				N		
T300/934	Bohlmann-Dergy [1]	N							
	Reinhart [2]	N							
T300/5208	McKague et al [3]	L							
	Kibler [4]	L							
	Augl [5]					N			
	Lundemo-Thor [6]		S						L
T300/5209	Stoecklin* [7]		S	N	N	S			
T300/2544	Stoecklin* [7]		S	S	S	S			
T400/2544	Trabocco-Stander* [8]		N-L	S	N				
AS/3501	Stoecklin* [7]		S	S	N				
	Trabocco-Stander* [8]			N					
AS/3501-5	Delsai-Whiteside [9]	N							
AS/X-2546	Browning-Hartness [10]					L			
HMS/339	Camahort et al [11]	N							
HMS/934	Camahort et al [11]	S					N		
HMS6759	Camahort et al [11]	S					N		
HMS/3501	Camahort et al [11]	N						N	
HMS/X-2546	Browning-Hartness [10]							N	
HTS/3002	Trabocco-Stander* [8]				N	L			
HTS/4617	Browning-Hartness [10]		N	N	S				
HTS/ADX 516	Browning-Hartness [10]		N	N	S				
HTS/P 13 N	Browning-Hartness [10]		N	N	S				
HTS/X-2546	Browning-Hartness [10]		N	L					
Modmor II/5206	Trabocco-Stander* [8]				N	L			
Narmco 2387(nr)	Browning-Hartness [10]		L						
ERL 2256(nr)	Browning-Hartness [10]		S						
ERLA 4617(nr)	Browning-Hartness [10]		L						
X2546(nr)	Browning-Hartness [10]		L						

N – negligible effect; S – small effect; L – large effect; (nr) neat resin; * – weathering test

NOTE: Numbers in square brackets refer to literature in [198]

TABLE 2

SUMMARY OF EXPERIMENTAL DATA ON THE EFFECTS OF MOISTURE AND TEMPERATURE ON THE ULTIMATE TENSILE STRENGTH OF COMPOSITES[261]

Composite	*Reference*	*Laminate Lay-Up Orientation*						*Remarks*
		0°		*π/4*		*90°*		
		Moist	*Temp*	*Moist*	*Temp*	*Moist*	*Temp*	
Thornel 300/Fiberite 1034	Shen & Springer, 1976	L	N	L	N	S	S	
Hercules AS-5/3501	Browning et al, 1976 [3]	N	N	N	N	S	S	
	Verette, 1975 [4]	N	N	N	–	S	S	Limited data (2–3 points)
	Kerr, et al, 1975 [5]	–	N	–	N	–	S	Two data points for 90° laminates
	Kim & Whitney, 1976 [6]	–	–	N	N	–	–	
Thornel 300/Narmco 5208	Hofer et al, 1975 [7]	L	L	N	L	S	S	
	Husman, 1976 [8]	–	–	–	–	S	L	
Modmor II/Narmco 5206	Hofer et al, 1974 [9]	N	L	N	L	S	S	
Courtaulds HMS/Hercules 3002M	Hofer et al, 1974 [9]	N	N	N	N	S	S	Very scattered data for 90° laminates
HT-S/ERLA-4617	Browning, 1972 [10]	–	–	L	S	–	–	Only two data points for temperature
HT-S/Fiberite X-911	Browning, 1972 [10]	–	–	N	N	–	–	
HT-S/U.C.C.X-2546	Browning, 1972 [10]	–	–	L	N	–	–	
PRD 49/ERLB-4617	Hanson, 1972 [11]	–	L	–	–	–	–	
HT-S/(8183/137-NDA-BF$_3$:MEA)	Hertz, 1973 [12]	–	–	–	–	S	S	
HT-S/Hysol ADX-516	Browning, 1972 [10]	–	–	N	S	–	–	Only two data points for temperature
Hercules HT-S/710 Polyimide	Kerr, et al, 1975 [5]	–	N	–	N	–	N	Only two data points for 90° laminates
HT-S/P13N Polyimide	Browning, 1972 [10]	–	–	–	L	–	–	
Boron/AVCO 5505	Hofer, et al, 1974 [9]	L	N	L	L	S	S	
Boron/Narmco 5505	Kaminski, 1973 [13]	–	L	–	–	–	S	
	Browning, 1972 [10]	–	–	N	N	–	–	

(a) N = Negligible effect (b) L = Little effect (< 30%) (c) S = Strong effect (> 30%)

NOTE: Numbers in square brackets refer to literature in [198]

TABLE 3

SUMMARY OF EXPERIMENTAL DATA ON THE EFFECTS OF MOISTURE AND TEMPERATURE ON THE ELASTIC MODULUS OF COMPOSITE MATERIALS[260]

Composite	Reference	Laminate Lay-Up Orientation					
		0°		π/4		90°	
		Moist	Temp	Moist	Temp	Moist	Temp
BUCKLING TEST							
Thornel 300/Fiberite 1034	Shen & Springer 1977	N	N	N	N	S	S
TENSILE TEST							
Hercules AS-5/3501	Browning, et al 1976 [5]	L	N	L	N	S	S
	Verette 1975 [6]	N	N	N	-	S	S
	Kerr et al 1975 [7]	-	N	-	N	-	-
Thornel 300/Narmco 5208	Hofer et al 1975 [8]	N	N	N	N	N	N
	Husman 1976 [9]	-	-	-	-	S	S
Modmor II/Narmco 5206	Hofer et al 1974 [10]	N	N	N	N	S	S
Courtaulds HMS/Hercules 3002M	Hofer et al 1974 [10]	N	N	N	N	N	S
HT-S/ERLA-4617	Browning 1972 [11]	-	-	N	S	-	-
HT-S/Fiberite X-911	Browning 1972 [11]	-	-	N	N	-	-
HT-S/UCC X-2546	Browning 1972 [11]	-	-	N	L	-	-
PRD-49/ERLB-4617	Hanson 1972 [12]	-	S	-	-	-	-
HT-S/(8183/137-NDA-BF_3: MEA)	Hertz 1973 [13]	-	-	-	-	N	S
TENSILE TEST							
HT-S/Hysol ADX-516	Browning 1972 [11]	-	-	N	S	-	-
HT-S/710 Polyimide	Kerr et al 1975 [7]	-	N	-	N	-	-
HT-S/P13N Polyimide	Browning 1972 [11]	-	-	-	L	-	-
Boron/WCO 5505	Hofer et al 1974 [10]	N	N	N	N	S	S
Boron/Narmco 5505	Browning 1972 [11]	-	-	N	N	-	-
COMPRESSIVE TEST							
Hercules AS-5/3501	Verette 1975 [6]	N	N	-	-	L	S
Thornel 300/Narmco 5208	Hofer et al 1975 [8]	L	N	N	N	L	N
Modmor II/Normco 5206	Hofer et al 1974 [10]	N	N	N	N	S	S
Courtaulds HMS/Hercules 3002M	Hofer et al 1974 [10]	N	N	N	N	S	S
Boron/WCO 5505	Hofer et al 1974 [10]	N	N	N	N	S	S

a) N = Negligible effect

b) L = Little effect (<30%)

c) S = Strong effect (>30%)

NOTE: Numbers in square brackets refer to literature in [260]

TABLE 4

GRAPHITE/EPOXY TAPE AND FABRIC LAMINA DESIGN ALLOWABLES*[181]

LOAD DIRECTION	0° PROPERTY	MATERIAL	
		TAPE	FABRIC
TENSION	MODULUS - 10^6 PSI	20.5	9.8
	STRENGTH - 10^3 PSI	97.4	38.2
	STRAIN - 10^{-6} IN/IN	4750	3900
COMPRESSION	MODULUS - 10^6 PSI	18.5	8.7
	STRENGTH - 10^3 PSI	74.0	34.8
	STRAIN - 10^{-6} IN/IN	4000	4000

*WORST ENVIRONMENTAL CONDITION, 3/16 IN DIA NOTCH OR IMPACT DAMAGE

TABLE 5

STATIC SHEAR PROPERTIES OF THE COMPOSITE MATERIALS AFTER CONDITIONING[233]

Material	Treatment	Shear Strength MNm^{-2}	Shear Modulus GNm^{-2}	Shear Strain (x 10^{-3})
CFRP	As-received	76 ± 9 (8)	3.5 ± 0.6 (8)	39 ± 11 (8)
	Soaked	61 ± 6 (4)	3.4 ± 0.2 (4)	25 ± 10 (4)
	Dried	72 ± 9 (4)	3.5 ± 0.2 (4)	29 ± 4 (4)
	Annealed	81 ± 13 (4)	3.7 ± 0.6 (4)	37 ± 11 (4)
GRP	As-received	79 ± 3 (5)	3.8 ± 0.2 (3)	67 ± 4 (5)
	Soaked	55 ± 6 (4)	4.3 ± 0.2 (4)	40 ± 8 (4)
	Dried	63 ± 7 (4)	4.0 ± 0.4 (3)	31 ± 4 (4)
	Annealed	82 ± 2 (3)	3.5 ± 0.3 (3)	37 ± 4 (3)
KFRP	As-received	48 ± 4 (6)	1.5 ± 0.1 (6)	27 ± 3 (6)
	Soaked	27 ± 2 (4)	1.4 ± 0.3 (4)	34 ± 1 (4)
	Dried	33 ± 2 (3)	1.3 ± 0.1 (4)	31 ± 5 (4)
	Annealed	34 ± 2 (4)	1.4 ± 0.1 (4)	27 ± 1 (3)

Uncertainties are standard deviations

Figures in brackets are the number of specimens

TABLE 6

RESULTS OF EXPOSURE ABOARD US CONSTELLATION AND NIMITZ CARRIERS OF AS/3501-6 MATERIAL[294]

	Unpainted Unexposed	US CONSTELLATION Exposure	NIMITZ * Exposure
Tensile Strength			
P.S.I.			
R.T.	55831	62922	58821
82.2°C	57432	61674	63292
121.1°C	61016	60111	57767
Shear Strength			
P.S.I.			
R.T.	6257	6149	6015
82.2°C	5964	6157	6807
121.1°C	6108	5920	4276
Flexural Strength			
P.S.I.			
R.T.	53232	52178	56489
82.2°C	52389	47253	59933
121.1°C	53544	46164	49191

* Nuclear Carrier

TABLE 7

SUMMARY OF MECHANICAL PROPERTIES OF SP-250/S-2 FIBERGLASS EPOXY[185]

Fabrication	Layup: 0^{o}, quasiisotropic		Pressure: 50 PSI	Cure: 30 min/250F		Post Cured: 90 min/250F		Plies: 8,12,16
Physical Properties	Weight % Resin: 50.1		Density: 1.85	Avg. % voids 0.53		Avg. Thickness: 0.009 in/ply		
Test Methods	Tension: ASTM D-3039			Compression: ASTM D-3410		Interlaminar Shear: ASTM D-2344		
Temperature	75F						160F	
Condition	0% RH 1 Month		50% RH 3 Months		95% RH 3 Months		95% RH 1 Month	
	Avg	SD	Avg	SD	Avg	SD	Avg	SD
Tension, QI*								
ultimate stress, ksi	74.32	4.94	75.56	3.00	62.87	3.42	48.26	3.09
ultimate strain, %	3.48	0.44	3.61	0.19	2.75	0.06	2.07	0.13
lower modulus, 10^6 psi	3.50	0.28	3.22	0.13	3.31	0.18	3.34	0.18
upper modulus, 10^6 psi	1.80	0.15	1.77	0.10	1.89	0.15	1.93	0.10
secant modulus, 10^6 psi	2.12	0.14	2.15	0.15	2.26	0.14	2.32	0.10
Tension, 0^{o}								
ultimate stress, ksi	228.62	16.00	222.90	15.43	174.25	13.20	141.53	14.16
ultimate strain, %	3.31	0.43	3.55	0.17	2.78	0.12	2.25	0.07
secant modulus, 10^6 psi	6.43	0.56	6.37	0.31	6.41	0.37	6.49	0.25
Compression, QI**								
ultimate stress, ksi	73.11	3.73	72.75	3.47	67.81	3.56	62.76	3.88
ultimate strain, %	2.96	0.34	3.06	0.13	2.73	0.23	3.10	0.35
secant modulus, 10^6 psi	2.55	0.19	2.49	0.30	2.31	0.18	2.31	0.50
Interlaminar Shear, 0^{o}								
ultimate stress, ksi	9.72	0.76	9.38	0.64	9.44	0.67	7.74	0.60

* $(0/+45/-45/90)_s$

** $(0/+45/-45/90)_{2s}$

TABLE 8

FLEXURAL TESTS ON E-2A ROTODOME, SERIAL NO. 1 AT 25°C[199]

TEST AREA	AGED NORMALIZED (0.011" PLY)				ORIGINAL DATA (POLAR PLOTS)			
	STR.		MOD.		STR.		MOD.	
	KSI	MPa	MSI	GPa	KSI	MPa	MSI	GPa
UPPER SKIN - PAINTED	53.8	371	-	-	61.5	424	-	-
	56.6	390	-	-	63.5	438	-	-
	57.0	393	-	-	63.5	438	-	-
AVG.	55.8	384	-	-	62.8	433	-	-
PERCENT RETENTION	89		-		-		-	
	KSI	MPa	MSI	GPa	KSI	MPa	MSI	GPa
BOTTOM SKIN - PAINTED	76.5	526	2.34	16.1	64.0	441	2.75	18.9
	65.9	545	2.37	16.3	57.5	396	2.30	15.8
	76.4	526	2.37	16.3	62.0	427	2.50	17.2
AVG.	72.9	502	2.36	16.2	61.2	422	2.52	17.4
PERCENT RETENTION	100+		93.7		-		-	
	KSI	MPa	MSI	GPa	KSI	MPa	MSI	GPa
UPPER SKIN - NO PAINT	57.2	394	1.88	13.0	63.0	434	1.9	13.1
	59.6	411	1.88	13.0	66.0	455	2.0	13.8
	59.9	413	1.90	13.0	67.3	464	2.1	14.5
AVG.	58.9	406	1.89	13.0	65.9	454	2.0	13.8
PERCENT RETENTION	89.4		94.5		-		-	
	KSI	MPa	MSI	GPa	KSI	MPa	MSI	GPa
INNER SKIN - NO PAINT MOISTURE CONTENT:1.1%	53.5	369	1.36	9.4	61.5	424	1.8	12.4
	56.3	391	1.45	10.0	59.0	407	1.9	13.1
	57.3	395	1.63	11.2	60.0	413	1.7	11.7
AVG.	55.9	385	1.48	10.2	60.2	415	1.8	12.4
PERCENT RETENTION	92.8		82.2		-		-	
CAP - NO PAINT, ERODED MOISTURE CONTENT: 0.24%	43.9	302	-		65.0	488	-	-
PERCENT RETENTION	67.5		-		-		-	-

TABLE 9

TENSILE TESTS ON E-2A ROTODOME, SERIAL NO. 1, 25°C[199]

TEST AREA	AGED NORMALIZED (0.011"/PLY)				ORIGINAL DATA (POLAR PLOTS)			
	Strength		Modulus		Str. KSI		MOD, MSI	
	KSI	MPa	MSI	GPa	KSI		MSI	
Bottom Skin - Painted								
Moisture	37.2	256	2.07	14.3	32.0	220	2.1	14.4
Content: 1.32%	25.3	174	1.98	13.6	32.0	220	1.7	11.7
	34.4	237	1.94	13.4	31.5	215	2.2	15.2
	35.1	242	2.89	13.0	36.5	251	2.3	15.8
	37.8	260	1.88	13.0	36.5	251	2.3	15.8
AVG.	34.0	234	1.95	13.4	33.7	232	2.1	14.4
Percent Retention	100+		92.9					
Upper Skin Painted								
Moisture	39.5	272	1.83	12.6	36.3	250	2.3	15.8
Content: 1.46%	42.9	296	2.09	14.4	39.4	271	2.5	17.2
	43.5	300	2.27	15.6	38.4	265	2.6	17.9
AVG.	42.0	289	2.06	14.2	38.0	262	2.5	17.2
Percent Retention	100+		82.4					
Upper Skin No Paint								
Moisture	35.0	241	1.77	12.2	37.0	255	2.5	17.2
Content: 1.00%	34.0	234	1.88	13.0	40.0	276	2.8	19.3
	38.0	265	1.94	13.4	39.0	269	2.5	17.2
	32.3	223	1.74	12.0	40.0	276	2.5	17.2
	38.5	265	1.98	13.6	40.0	276	2.7	18.6
AVG.	35.7	246	1.86	12.8	39.0	269	2.6	17.9
Percent Retention	91.5		71.5					

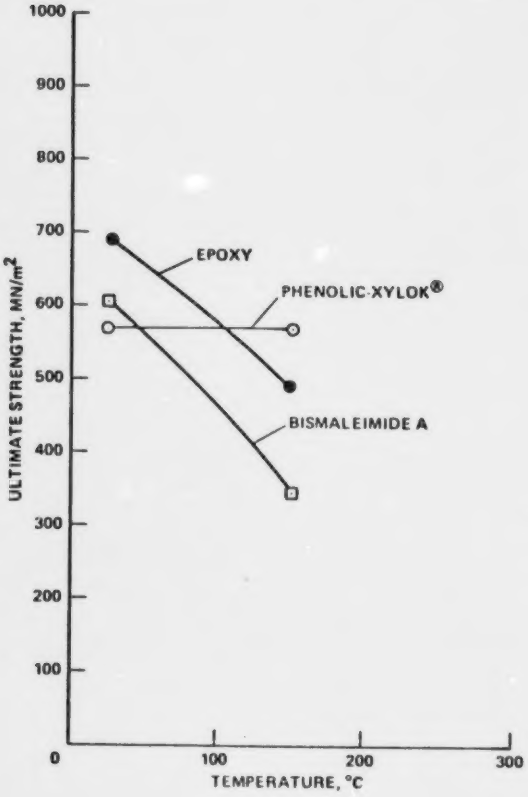


FIG. 1: EFFECT OF TEMPERATURE ON TENSILE STRENGTH OF GRAPHITE COMPOSITES[178]

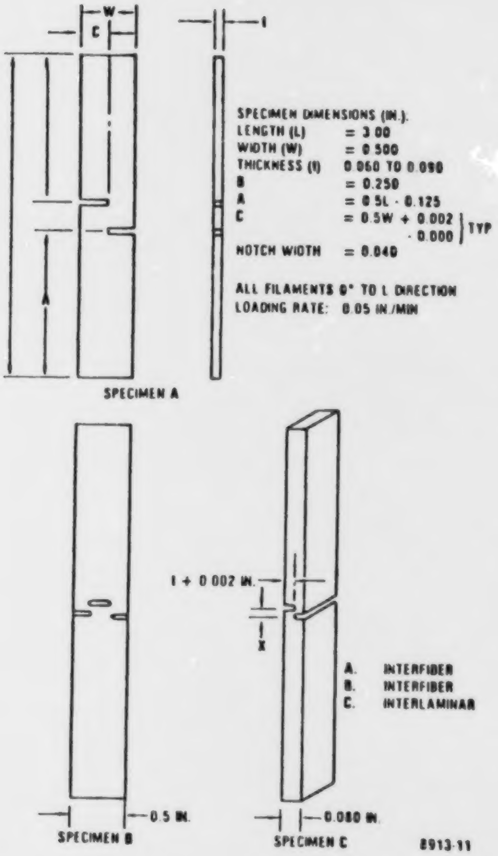

W
C
t
A
SPECIMEN DIMENSIONS (IN.).
LENGTH (L) = 3.00
WIDTH (W) = 0.500
THICKNESS (t) 0.060 TO 0.090
B = 0.250
A = 0.5L - 0.125
C = 0.5W + 0.002 - 0.000 TYP
NOTCH WIDTH = 0.040
ALL FILAMENTS 0° TO L DIRECTION
LOADING RATE: 0.05 IN./MIN
SPECIMEN A
t + 0.002 IN.
X
A. INTERFIBER
B. INTERFIBER
C. INTERLAMINAR
0.5 IN.
SPECIMEN B
0.080 IN.
SPECIMEN C
8913-11

FIG. 2: SHEAR TEST SPECIMEN[5]

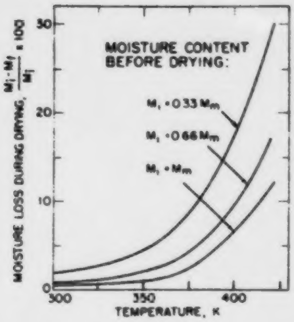


FIG. 3: MOISTURE LOSS OF SPECIMEN DURING THREE MINUTES OF DRYING AT DIFFERENT TEMPERATURES. M_i AND M_f ARE THE MOISTURE CONTENTS BEFORE AND AFTER DRYING. M_m DENOTES MOISTURE CONTENT AT FULL SATURATION[261]

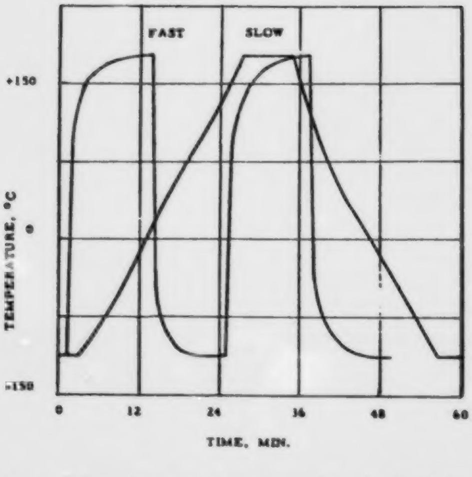


FIG. 4: SCHEMATIC OF THERMAL CYCLES FOR TEMPERATURE CYCLING SYSTEMS[209]

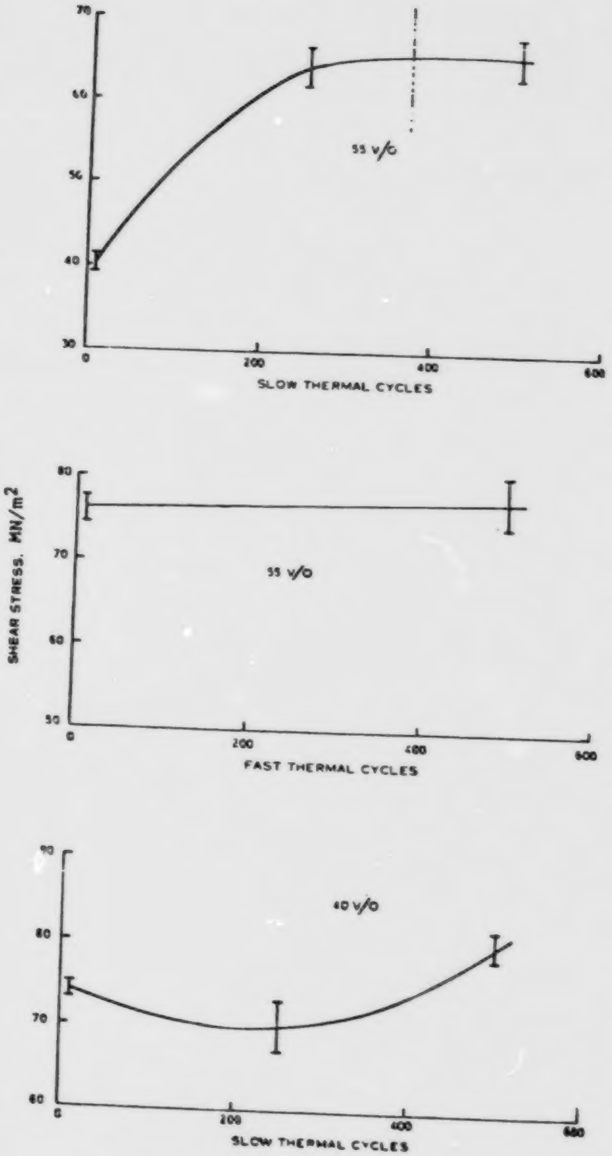


FIG. 5: SHEAR STRENGTH OF CARBON/EPOXY COMPOSITES AS A FUNCTION OF THERMAL CYCLING[209]

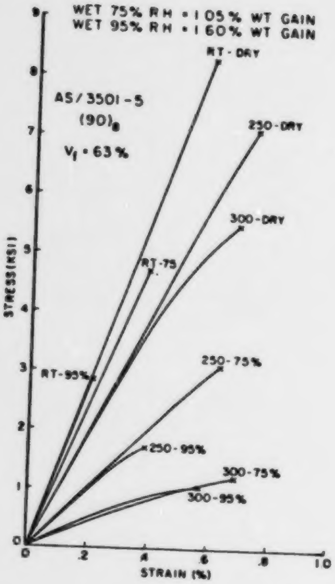


FIG. 6: TYPICAL TRANSVERSE STRESS-STRAIN CURVES AS A FUNCTION OF TEMPERATURE FOR AS/3501-5 UNIDIRECTIONAL COMPOSITES[41]

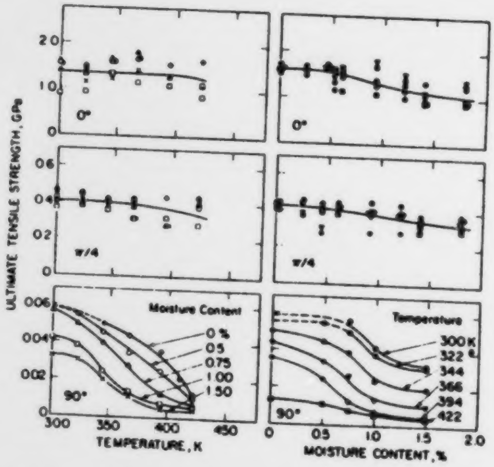


FIG. 7: ULTIMATE TENSILE STRENGTH OF THORNEL 300/FIBERITE 1034 AS A FUNCTION OF TEMPERATURE AND MOISTURE CONTENT. PRESENT DATA. FIBER VOLUME FRACTION ~0.68. 1GPa = 1.450 X 10^5 lbf/in^2[261]

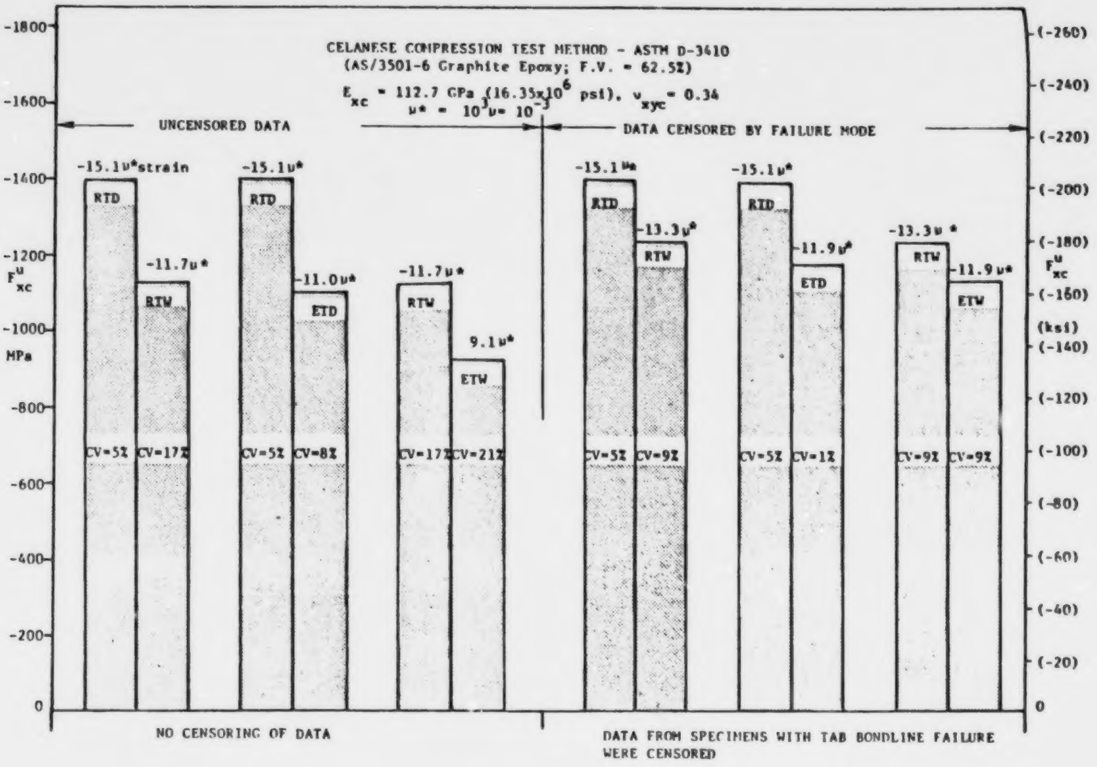


FIG. 8: 0-DEGREE STATIC COMPRESSION STRENGTH OF LAMINATE A, $[0]_{16T}$[115]

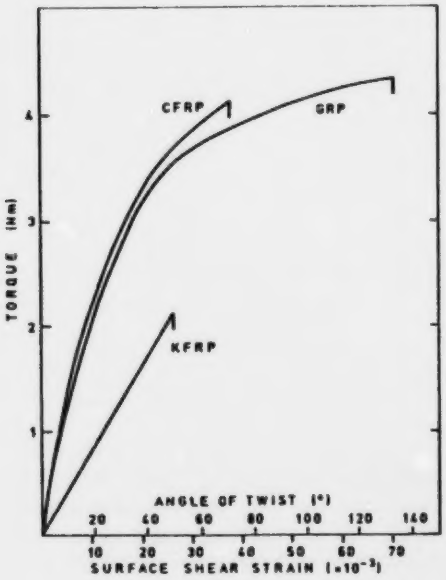


FIG. 9: TORQUE-TWIST BEHAVIOR OF THE AS-RECEIVED COMPOSITES[233]

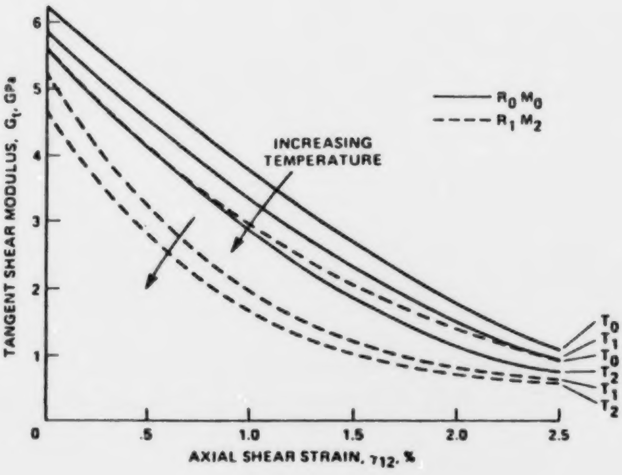


FIG. 10: TEMPERATURE EFFECTS ON TANGENT SHEAR MODULUS[193]

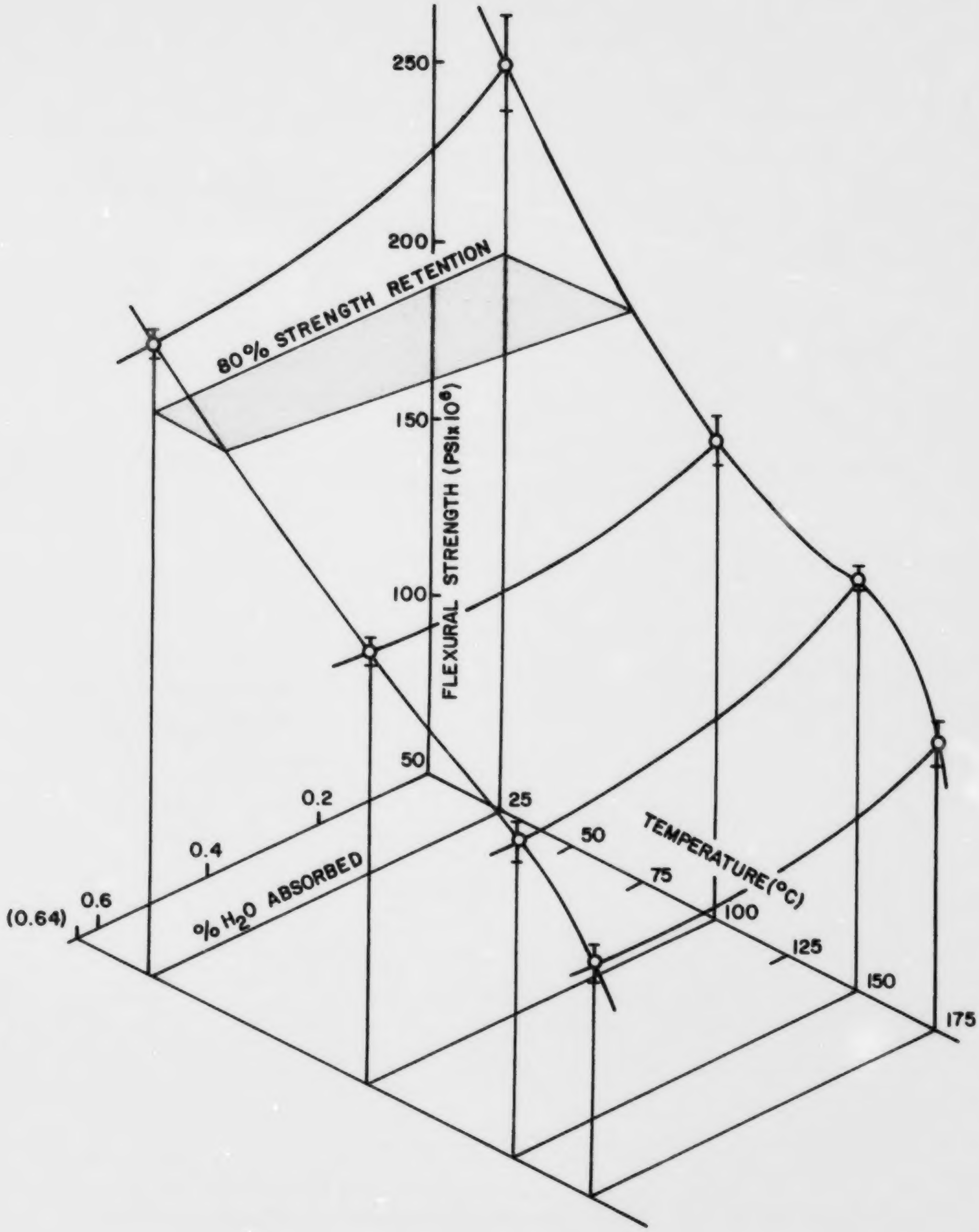


FIG. 11: FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (NARMCO 5208/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED[23]

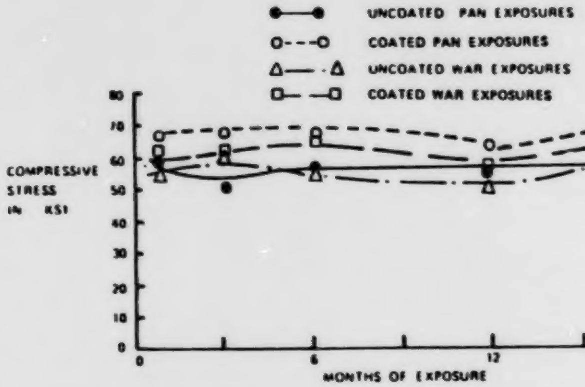


FIG. 12: THE EFFECT OF EXPOSURE TIME ON COMPRESSIVE STRENGTH OF 3002T GRAPHITE/EPOXY[291]

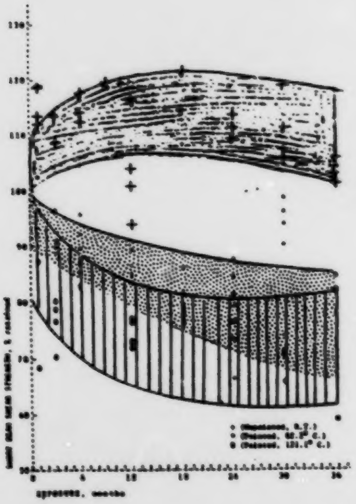


FIG. 13: 400 SERIES, 10 PLY (0/±45/90) WARMINSTER, % RETAINED SHORT BEAM SHEAR STRENGTH, AS/3501-6 GRAPHITE/EPOXY[294]

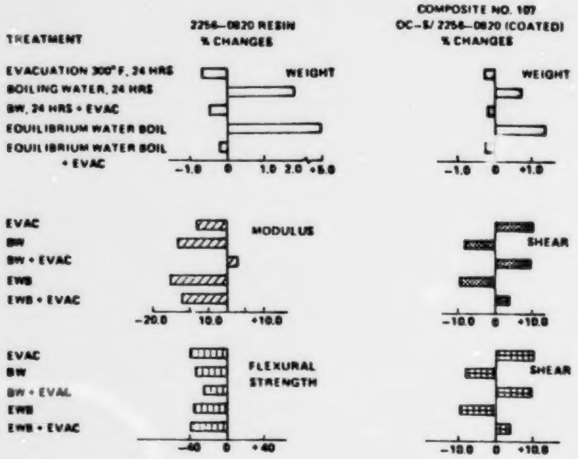


FIG. 14: WATER EFFECTS ON RESIN AND COMPOSITE[255]

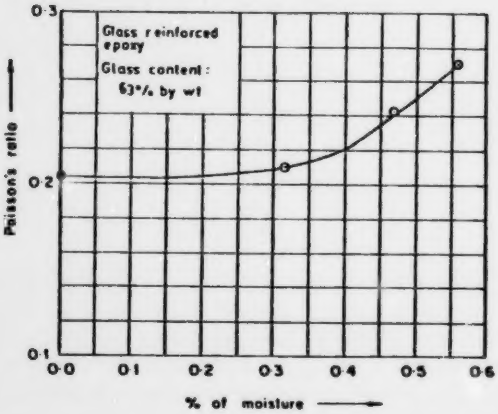


FIG. 15: EFFECT OF MOISTURE ON THE POISSON'S RATIO OF A GLASS CLOTH REINFORCED EPOXY COMPOSITE[238]

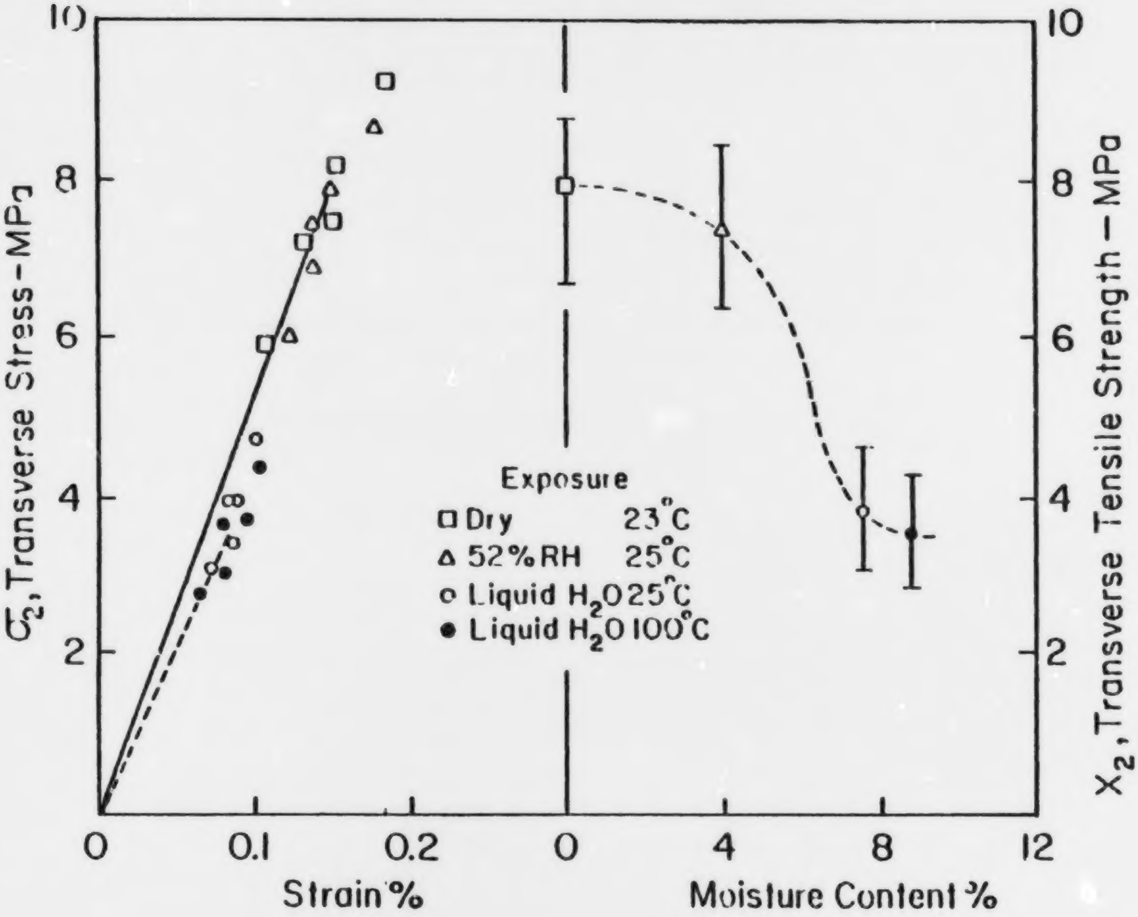


FIG. 16: TRANSVERSE TENSILE BEHAVIOR OF K-49 EPOXY AT DIFFERENT ENVIRONMENTAL CONDITIONS. LEFT, AVERAGE STRESS-STRAIN RESPONSES AND FAILURE POINTS; RIGHT, TRANSVERSE TENSILE STRENGTH AS A FUNCTION OF MOISTURE CONTENTS (MPa X 0.145 = Ksi)[322]

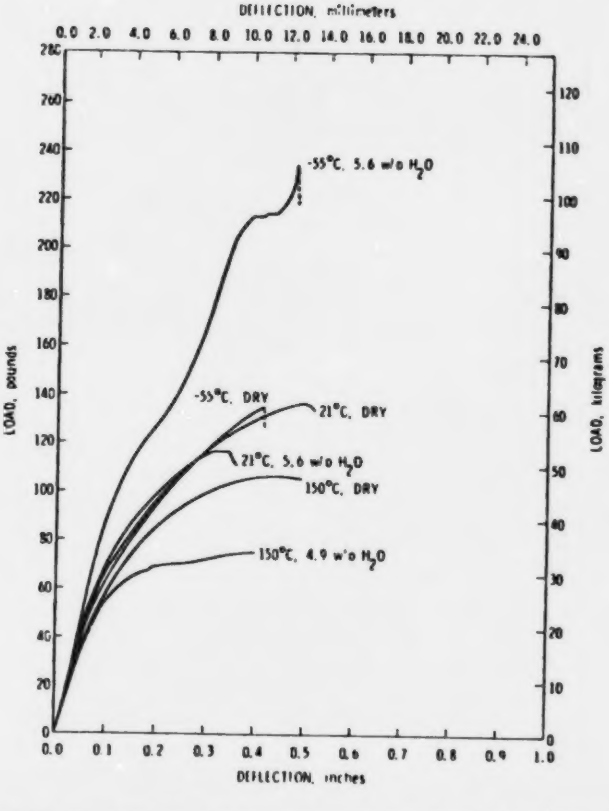
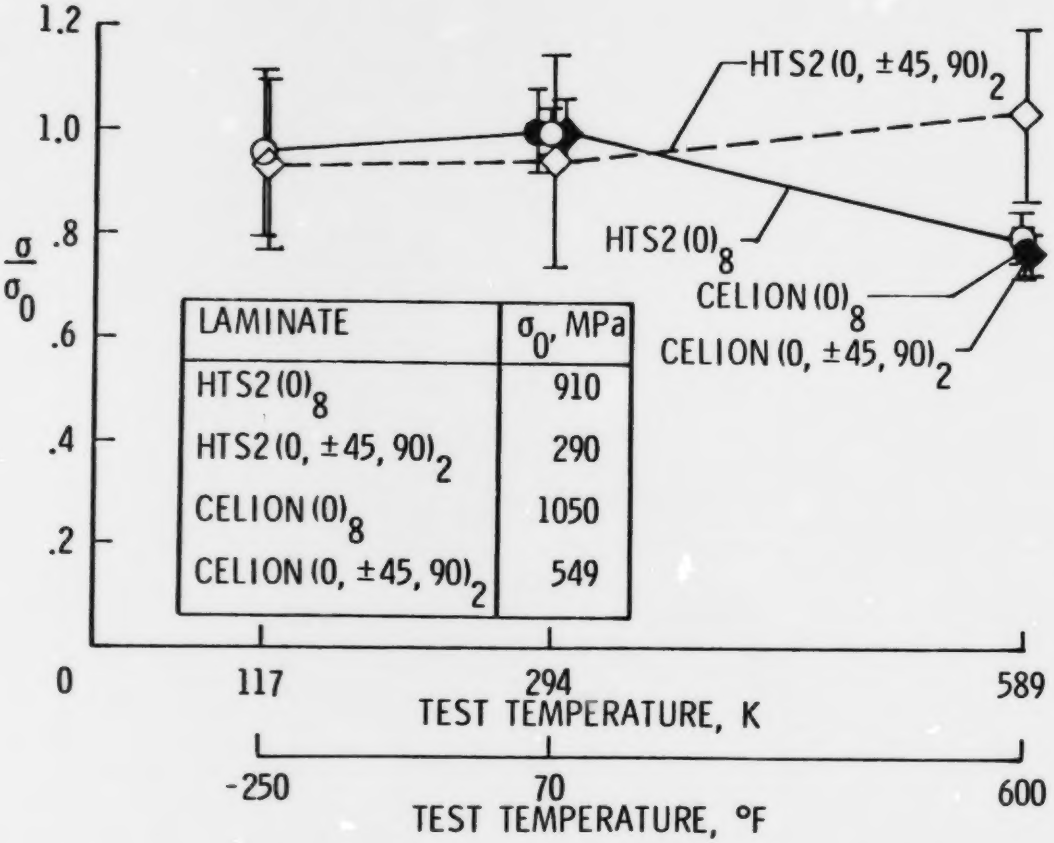
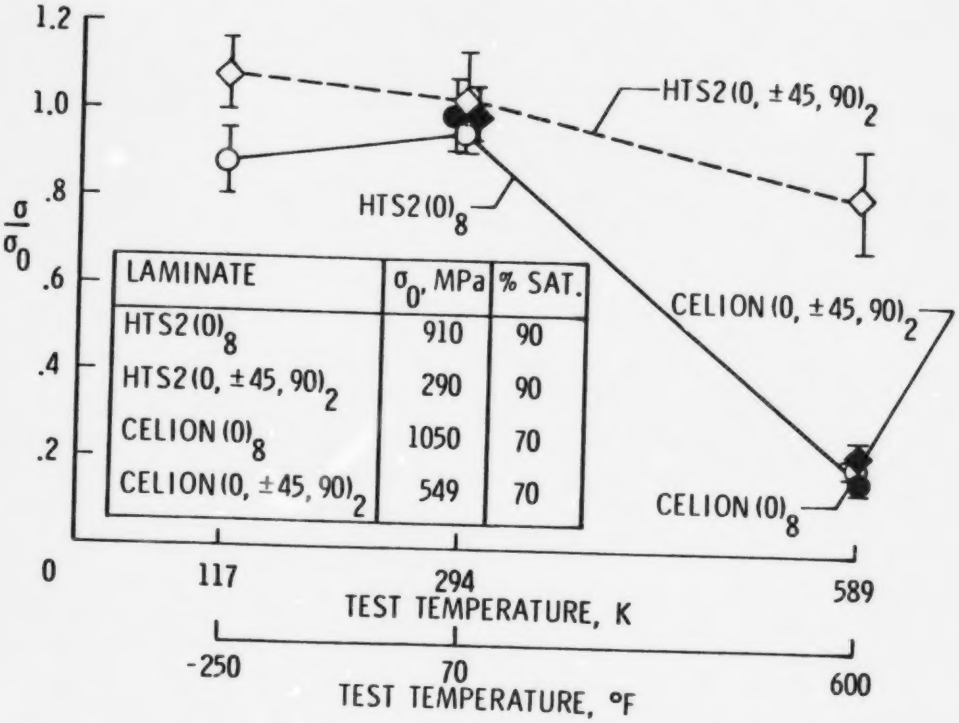


FIG. 17: LOAD VERSUS DEFLECTION BEHAVIOR FOR [0/90] KEVLAR 49 181 STYLE FABRIC/CE-9000 EPOXY LAMINATES AS A FUNCTION OF TEMPERATURE AND MOISTURE CONTENT[10]



LAMINATE	σ_0, MPa
HTS2 (0)$_8$	910
HTS2 (0, ±45, 90)$_2$	290
CELION (0)$_8$	1050
CELION (0, ±45, 90)$_2$	549

FIG. 18: FLEXURAL STRENGTH OF DESSICATED SPECIMENS[104]



LAMINATE	σ_0, MPa	% SAT.
HTS2(0)$_8$	910	90
HTS2(0, ±45, 90)$_2$	290	90
CELION(0)$_8$	1050	70
CELION(0, ±45, 90)$_2$	549	70

FIG. 19: FLEXURAL STRENGTH OF "WET" SPECIMENS[194]

APPENDIX A — BIBLIOGRAPHY

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J.P. Komorowski

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SUMMARY/SOMMAIRE

This is Part III of a series of literature reviews on hygrothermal effects on polymer matrix composite materials. It contains a review of papers on mechanical properties as measured in static tests and includes the effect of impact damage interaction with environmental conditions.

The other parts of the review are:

Part I:	Moisture and Thermal Diffusion
Part II:	Physical Properties
Part IV:	Mechanical Properties 2
Part V:	Composite Structures and Joints
Part VI:	Numerical and Analytical Solutions
Part VII:	Summary of Conclusions and Recommendations

A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.

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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART IV: MECHANICAL PROPERTIES 2 FATIGUE AND TIME-DEPENDENT PROPERTIES

by

J. P. Komorowski

National Aeronautical Establishment

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PART IV: MECHANICAL PROPERTIES 2 FATIGUE AND TIME-DEPENDENT PROPERTIES

EFFETS HYGROTHERMIQUES DANS LES COMPOSITES À RENFORT DE FIBRE CONTINU

PARTIE IV: PROPRIÉTÉS MÉCANIQUES 2 FATIGUE ET PROPRIÉTÉS DÉPENDANT DU TEMPS

by/par

J.P. Komorowski

National Aeronautical Establishment

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W. Wallace, Head/Chef
Structures and Materials Laboratory/
Laboratoire des structures et matériaux

G.M. Lindberg
Director/Directeur

SUMMARY

This is part IV of a series of literature reviews on hygrothermal effects on polymer matrix composite materials. It contains a review of papers on mechanical properties as measured in fatigue, creep or stress relaxation tests with variations in temperature and humidity accounted for in the results.

The other parts of the review are:

Part I: Moisture and Thermal Diffusion
Part II: Physical Properties
Part III: Mechanical Properties 1
Part V: Composite Structures and Joints
Part VI: Numerical and Analytical Solutions
Part VII: Summary of Conclusions and Recommendations

A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.

RÉSUMÉ

Voici la partie IV d'une série de documents traitant des effets hygrothermiques sur les matériaux composites à matrice polymérique. Elle comprend une étude des données recueillies sur leurs propriétés mécaniques lors d'essais de fatigue, de fluage et de relâchement des contraintes dont les résultats tiennent compte des variations de température et d'humdité.

Les autres parties de cette série sont les suivantes:

Partie I: Diffusion de l'humidité et de la chaleur
Partie II: Propriétés physiques
Partie III: Propriétés mécaniques 1
Partie V: Structures et joints composites
Partie VI: Solution numériques et analytiques
Partie VII: Résumé des conclusions et recommandations

Une liste complète des références est incluse en annexe et les nombres entre parenthèses dans le texte se rapportent à cette liste.

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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART IV: MECHANICAL PROPERTIES 2 – FATIGUE AND TIME-DEPENDENT PROPERTIES

1.0 INTRODUCTION

In this part of the review (Part IV) results of fatigue and creep and stress-relaxation tests are reported.

With the move towards primary load carrying structures made with composite materials, the number of publications related to the fatigue properties of these materials has rapidly increased. Most of these papers are concerned with graphite/epoxies, and with other materials receiving less attention either because of cost or inferior properties. In the future, if the trend towards higher strain allowables for structural composite materials is realized, fatigue will certainly become a major factor in the design of primary composite structures.

There is a strong indication that fatigue in composites is closely related to creep as time at load, rather then number of cycles, has had the greater effect on life in some experiments. Mechanical relaxation problems, however, have significance of their own for some applications like pressure vessels, bolted joints (relaxation of clamp up force) or structures which have to demonstrate dimensional stability (relaxation of residual stresses).

Fatigue tests of composite structures and joints will be reviewed in Part V.

2.0 FATIGUE

2.1 Constant Amplitude Loading Studies

Constant amplitude loading studies that produce S-N curves have limited value for composite designers. They are, however, a very convenient method of demonstrating behavior of materials under cyclic loading and have been used in environmental studies of composite materials. In this chapter investigations will be reviewed in which materials have been exposed to various conditions of temperature and humidity prior to, during or after constant amplitude cycling.

Hofer, Bennett and Stander[131] studied the effect of humidity preconditioning followed by stress cycling, on residual mechanical properties. Earlier in the study S-N curves were generated at room temperature. The stress ranges at 2×10^6 cycles were taken from these curves for all laminates under consideration. The average stress range was calculated and a 10% smaller value was used as stress range for stress cycling. Humidity preconditioning consisted of steady state 500 hour or 1000 hour soaking at 48.9°C – 98 ± 2% RH. Materials used were T300 (graphite) and S-glass in 5208 epoxy resin. Hybrid laminates of an interweaving type were made with varying proportions of all glass and all graphite plies. Lay-up was either unidirectional or quasi-isotropic. All materials were stressed in a tension-tension load cycle ($R = 0.1$) at 30 Hz up to 10^7 cycles but not all specimens survived this rather mild stress cycling regime. Those that did showed a decrease in residual strength but no loss of modulus and slight increase in Poisson ratio for the 0° lay-ups.

Rotem and Nelson[245] and Rotem[246] studied specimens of graphite/epoxy (T300/5208) in tension-tension ($R = 0.1$ and $R = 10$ at 30 Hz) at 22°C, 74°C and 114°C. They used unidirectional and angle-ply laminates as well as $[0°/\pm\theta/0°]_S$. These latter laminates were used to verify predictions of fatigue durability and failure mode based on results for unidirectional and angle-ply laminates. The fatigue behavior of a single lamina was characterized by its static strength and its "fatique function" which expresses the degradation in static strength due to cyclic loading. This function measured at some reference temperature together with temperature shifting factors can be used to calculate the fatigue function in a broad range of temperatures. Results generally correlated well with predictions but for laminates where angle-ply laminae contributed to the load to a greater extent ($\theta < 45°$), the viscoelastic character of the matrix had to be taken into account. Generally for matrix dominated laminates, fatigue failure was affected by cycling and temperature, and a shifting of the fatigue function was observed. However, the slope of the S-N curve was not affected by temperature. Fiber dominated laminates were not sensitive to temperature change (Figs. 1, 2, 3 and 4).

Haskins, Kerr and Stein[127] presented results for HT-S/710 graphite/polyimide in $[0°/\pm45°]_S$ lay-up. Fatigue tests were carried out at RT and 232°C. For the two loading ratios ($R = -1$, $R = 0.1$), there was little effect of temperature on fatigue of this fiber dominated laminate which supports the results of Rotem and Nelson.

Kan and Ratwani[161] presented results for matrix dominated laminates $(\pm45/90_2/\pm45/90_2)_S$ made from graphite/epoxy (AS/3501-6). Specimens were moisture preconditioned up to 1% weight gain with the uniform moisture distribution calculated using Fick's model as defined by Springer and Shen[212]. Fatigue tests were run under tension-compression fully reversed loading ($R = -1$). Test frequency of 5 Hz was chosen as the final loading frequency. Figure 5 shows the influence of moisture content on the fatigue behavior of the laminate. It was tentatively concluded that for this matrix dominated laminate, at room temperature, moisture had no influence on the compression fatigue life.

It would appear from all of the above reviewed results, that temperature is more detrimental than moisture, in reducing fatigue strength.

Ryder and Walker[251] have done an extensive study of the effect of compressive loading on the fatigue of graphite/epoxy (T300/934) laminates. The aim was to observe the effect of absorbed moisture under compressive loading in unnotched and notched (circular hole) specimens. Two lay-ups were used (1) 25% of 0° $-(0/45/90/-45_2/90/45/0)_S$, (2) 67% of 0° $-(0/45/0_2/-45/0_2/45/0_2/-45/0)_S$. The baseline dry condition was 22°C, 40 ± 10% RH while preconditioning took place at 82°C, 90% RH up to saturation for the wet tests. There are four ways of defining the loading variables in fatigue: load ratio (R), maximum stress (σ_{max}), stress range ($\Delta\sigma$) and minimum stress (σ_{min}). As $R = \sigma_{min}/\sigma_{max}$ and $\Delta\sigma = \sigma_{max} - \sigma_{min}$, during a fatigue test, any one of these variables may be held constant while the effect of one of the other three is being studied. Laminate (1) and (2) act as minimum columns. The maximum compressive stresses without lateral deflection greater than 0.0254 mm were therefore limited to −110 MPa and −207 MPa respectively. Constant load ratio was rendered impractical as it limited the maximum stresses. In these studies σ_{min} was held constant at either −110 MPa (−207 Ma) or 0(0). Failure was defined for tension-tension as breakage of coupon and for tension-compression as either breakage or an inability to sustain load due to severe delamination. Failure modes observed for elevated temperature, wet (ETW) conditions were similar to those obtained at RT. The only difference was that the type of damage which led to failure appeared much earlier in life for coupons tested at the same stress. For matrix dominated laminate (1) elevated temperature, wet conditions decreased life of unnotched specimens by a factor of 3, and for notched specimens by a factor of 10. For notched specimens, the tension-tension S-N curve changed from flat at RT to declining strength with number of cycles under ETW conditions. For the fiber dominated laminte (2) the results are not easy to discuss. A larger scatter was evident for ETW conditions. During tension-compression tests at RT, some specimens survived 10^6 cycles of +759 MPa stress, while under ETW, all specimens with stress above +550 MPa failed before 10^6 cycles were reached. The fatigue tests were followed by residual strength test and it was observed that the fatigue induced damage does not appear to have a direct effect on residual tensile strength. For laminate (2) cycled to 10^6 cycles under tension-tension ETW conditions, residual tensile strength was reduced by 20% while residual compressive strength was reduced by 40%. This indicates that ETW cycling has a significantly larger degrading effect than RT cycling which resulted in respective compressive strength reductions of 0% and 15%.

The data obtained was analyzed using Weibul distributions as well as other methods. Ryder and Walker concluded that significant statistical analysis efforts, combined with an extensive experimental investigation, is needed before any extrapolative procedures can be used with confidence to predict fatigue performance in an environmentally degraded condition.

The studies of the effect of moisture and temperature on composite material strength have indicated that compressive strength is particularly sensitive to these factors[187, 115]. Similarly compressive loads in fatigue have strong degrading effects on fatigue properties. However no evidence of a synergistic effect of compression fatigue and environment was found in the reviewed literature.

Grimes[115] carried out an investigation in which graphite/epoxy samples were loaded in compression-compression at R = 10. The material used was AS/3501-6 with the following lay-ups: $[0]_{nt}$, $[90]_{nt}$, $[\pm 45]_{nS}$ and $[(\pm 45)_5/0_{16}/90_4]$. Some samples were pre-soaked up to a 1.1% weight gain of moisture. Testing was carried out in a specially designed fixture which was used for both fatigue and residual strength tests. Fatigue testing was carried out at room temperature and the residual strength test was at an elevated (103.3°C) temperature. Only for $[90]_{24T}$ specimens were significant differences in fatigue properties found (Fig. 6). For dry samples, runouts were observed at stress levels of 126.9 Mpa (or 49% static dry strength) while for wet conditions, the runout stress level was 90 MPa (or 45% of static wet). However, these differences could be expected since for these samples, static dry strength is higher than static wet strength −258.6/−199.3 [MPa/MPa]. 'Wear out'* occurred in all specimens but was greater in matrix dominated laminates. Higher wearouts were attributed by Grimes to degradation of the interface.

Adams[3] reported results of an SEM study carried out on failed samples from Grimes' investigation. The influence of moisture or elevated temperature was not observed. No obvious differences were noted in the corresponding fracture surfaces. The author also tried to apply a micromechanics analysis which was developed earlier and successfully applied to calculate residual and environmental stresses. There is a similarity between static and fatigue failure in compression, however, the application of micromechanics analysis to fatigue is still far from being satisfactory.

Sumsion and Williams[277] and later Sumsion[278] studied the effects of temperature and water on flexural and torsional fatigue of AS/3501 graphite/epoxy laminates. The lay-ups used were 0°, ±45°, ±30° and woven (24 plies). Specimen shapes were as shown in Figure 7. Torsional fatigue tests were carried at 1 Hz under controlled strain (constant deflection) conditions and stopped if either the torque dropped to a preset level or if the required number of cycles was reached. Testing was carried out in air or water at both room and elevated (74°C) temperatures. After fatigue testing, the specimens were subjected to four point bending at room temperature to measure strength, and bending moment versus deflection curves were used to calculate failure energy. All the specimens exhibited fatigue damage. The effect of exposure to a water environment during torsion testing at 74°C and, to a lesser extent at 24°C was to decrease the 'incubation period'** and to increase the rate of accumulation of damage. At 74°C water also appeared to decrease (lower) the limiting torsional stiffness (Fig. 8). It should be noted that the cross plied specimens appeared more prone to fatigue damage in torsion than the unidirectional specimens. In contrast, in flexural fatigue, water had greater effect on unidirectional specimens.

The graphite/epoxy specimens showed significant flexural fatigue damage on both air and water when subjected to fully reversed plane bending at 30 Hz (Fig. 9).

Phillips, Scott and Buckley[233] also studied torsional fatigue of composites. The materials used were high modulus carbon, glass and Kevlar 49 in a Ciba-Geigy MY750 epoxy matrix cured with metyl nadic anhydride and benzyldimethylamine. A unidirectional lay-up was used and rods were machined to a 6 mm diameter. Test were run at room temperature and humidity. Samples were tested in either an as received state, or after seven days at 100°C water immersion. Some conditioned specimens were dried at 60°C for seven days prior to testing. Fatigue testing was carried out at 0.17 Hz, under either constant torque (±T) or constant twist ($\pm\theta$) amplitude. Significantly, the strength recovery upon drying observed during the static tests was not observed in torsional fatigue. Fatigue life for carbon and glass composites was permanently degraded by 100°C water immersion (Fig. 10). However, the boiling water test is very severe and is not similar to any situation encountered in service.

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* Observable damage.

** Time required to observe first damage of the material.

The rate of change of compliance has been measured in terms of ΔTorque/log N. For Kevlar composite, it is affected by various treatments as shown in Figure 11. In glass composites, this rate demonstrated partial recovery upon drying while in carbon composites, it was insensitive to moisture level.

Gauchel, Steg and Cowling[106] used Naval Ordinance Laboratory (NOL) ring samples filament wound using S-glass with various epoxy resins. Prior to fatigue testing in diametrical compression, rings were immersed at RT in water for 400 days. Testing was also carried out in water and concluded when the observed load at a given deflection dropped by 20%.

The percent retention of fatigue life after soak compared to dry specimens varied greatly (from 100% to 37.2%). The best results were achieved with a system containing 10 parts of N,N-diglycidyl tribromoaniline (DGTBA) for one part of meta-phenylenediamine (MPDS). Later tests showed that systems containing over 50% of DGTBA perform much better in fatigue under moisture influence than the other systems under consideration. These latter systems were mostly based on diglycidyl ether of bisphenol-A (DGEBA). The choice of resin may be detrimental to fatigue performance of a composite structure exposed to moisture.

In all the above papers the test conditions were steady state temperature and humidity, Lundemo[200, 201] studied the influence of environmental cycling on the static and fatigue properties of T300/5208, $(\pm 45)_{4S}$. The environmental cycles used prior to mechanical testing were aimed at simulating fighter aircraft service, including thermal spikes and low temperature excursions, with the humidities set to result in a moisture content of approximately 1% weight gain. Tension-tension ($R = 0.1$) fatigue tests were performed at $\sigma_{max} = \frac{1}{2}\,\sigma_{ultimate}$ and a frequency of 28 Hz. No failures occurred after 10^6 cycles for these specimens which were not exposed to the environmental treatment. After four weeks of treatment, five out of eight specimens survived 10^6 cycles with no survivals observed after six weeks of treatment (the longest life recorded for these specimens was 8.7×10^4 cycles. For specimens coated with polyurethane, less degradation was observed. Despite the low number of environmental cycles imposed (30 in six weeks) the effect was significant.

2.2 Random Loading

Years of experience has led to the conclusion that fatigue and damage tolerance testing must be conducted under conditions representative of service environments. Constant amplitude testing may be used only for those parts which will have a constant amplitude service environment. For the majority of aircraft parts this is not the case and simulation must be representative of the random nature of the service load history, usually with both amplitude and frequency variation. The flight-by-flight conditions including reverse loading must be represented[317]. This is true for both metal and composite structures. For composites, there is no accepted cumulative damage theory that allows the extrapolation of simple constant amplitude test data to the evaluation of the effects of random loading. Representative spectrum tests must be used and generally must start at the coupon level. This is expensive and time consuming, and by its nature, must be directed towards a particular application.

In a series of articles Haskins, Kerr, Stein et al[166, 126, 127] presented a program and some results on long term evaluation of Advanced Composites for Supersonic Cruise Aircraft. The materials used were graphite and boron/epoxy and polyimide (notably AS/3501, HT-S/710). Flight simulation was carried out using random loading with temperature cycling representative of supersonic flight (Fig. 12). Baseline tests and short term tests (accelerated load frequency and maximum temperature) were used to set reasonable load limits for long term tests. After 100 hours of testing, a preliminary wearout model was set and later refined after 200 test hours (Fig. 13). For the long term test, loads were set so that approximately 80% of the specimens would survive 25,000 simulated flights of two hours duration (one lifetime). During each flight one compressive load was applied at the highest temperature point of the flight. After 8000 hours, more specimen failures were observed than predicted from short term tests. It was concluded that the wearout model used is not sufficient to accurately predict complex real time exposure effects. Delamination was extensive as the failures were due to compression — which indicates that ultimate tensile strength is not a good measure of damage in composite.

Sendeckyj et al[258] studied the effect of temperature on fatigue response of surface-notched $[(0/\pm45/0)_S]_3$ T300/5208 graphite/epoxy. Flight-by-flight spectrum loading was used in two versions, one with and the other without compressive loads present. One lifetime consisted of 1280 flights, each 44 seconds in duration. Load cycling was carried out at different temperatures from 27°C to 210°C. All survivors of two lifetimes were inspected using both C-scan and TBE radiography and subjected to a room temperature residual tensile strength test. Elimination of compressive stresses did not lead to a significant change of residual strength. However, the fatigue test temperature did influence the residual strength. The maximum residual strength was observed for specimens tested between 156°C to 182°C. No specimen survived two lifetimes at 210°C but all had survived below that temperature. At increased temperatures, the size of the damaged zone increases (delamination) however, the more damage present the worse the conditions are for load transfer by the matrix. This, in turn, reduces the local stress intensity due to the presence of a crack (notch).

Results from the US Air Force Materials Laboratory sponsored Advanced Composites Serviceability Program have appeared in several papers[230, 12, 176, 177]. The main aim of this study was to develop experimental information on the growth of flaws and to quantify their influence on residual strength. A real time matrix of load, temperature and moisture was reduced and compressed. This permitted one lifetime to be simulated by 24 hours test. Loading was representative of a vertical tail spectrum (B1 bomber) including fully reversed load ($R = -1.0$). The maximum test load was equal to the design limit load (2/3 of ultimate allowable or 80% of average ultimate stress). Figure 14 contains a truncated spectrum for metal vertical tail with all cycles below a load factor (L.F.) of 0.089 removed (only the positive side of the $R = -1.0$ spectrum is shown). The final load and temperature spectrum can be seen in Figure 15. The number of load cycles was reduced from 500,000 to 127,500 and the number of temperature cycles was reduced from 4000 to 6 to enable one test lifetime to be carried out in 24 hours. The temperature sequence in a mission was rearranged into a monotonically increasing sequence from low to high. Preconditioning with moisture was carried out at 74°C, 98% RH up to a 1.2 to 1.3% weight gain to represent the worst type of USAF basing conditions. In order to maintain the moisture content, steam was injected into the system during the 49°C and 82°C cycles. Compromises made are summed up in Figure 16.

Specimens used in these studies were made of AS3501-5A and T300/5208. The flaws were classified into categories which describe the stress gradients caused by a flaw embodied in a laminate undergoing a far field (away from the flaw) uniform stress. The likelihood of occurrence was used to estimate the flaw criticality[177] and fatigue tests were carried out. The flaw size was regarded as critical if it led to specimen failure after two lifetimes of spectrum loading.

It was found that the residual compressive strength of graphite/epoxy, after load cycling degrades with temperature, moisture, proof loading and size of imperfection or damage, Figures 17, 18 and 19[176]. As delamination was the dominant damage type, residual tensile strength was not significantlly affected[177].

Daniel and Schramm et al[79, 253] conducted nondestructive inspections aimed at monitoring damage growth as part of the above described studies. They found that flaw growth was much more pronounced for those specimens exposed to environmental fluctuations in addition to the load spectrum. The worst type flaws appeared to be[79]:

1. Circular hole.
2. Embedded film patch.
3. Internal ply gap.
4. Surface scratches.

Gerharz and Schutz[108] presented a "quasi real time" program and proposed several accelerated schedules for testing the composite upper surface wing root area of a fighter plane. The load spectrum used was FALSTAFF*. The objective of the accelerated test program was to achieve the same damage growth and residual strength as found in a "real time" loading with a shorter testing time. To date no results of these studies have been published.

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* For more information see "Introduction to a Fighter Aircraft Loading Standard for Fatigue Evaluation "FALSTAFF" by G.M. vanDijk and J.B. deJonge.

When developing accelerated testing schedules the effect of creep must be considered. Sun and Chim[279] found that fatigue life increases with time at load. For notched samples of T300/5208 in a $[\pm 45]_{2S}$ lay-up, fatigue life was significantly longer when cycling frequency was first low and later higher. The reverse order of cycling frequency resulted in shorter fatigue life. They concluded that a reduction of stress intensity at a crack tip due to creep was responsible for increasing the fatigue life during the "slow-fast" tests. As creep is clearly related to temperature it is obvious that time at temperature in the environmental cycle will also influence fatigue life.

Other spectrum loading tests will be reviewed in Part V of this review which deals with composite joints and structures.

2.3 Fatigue Testing in Simulated Environmental Conditions

Testing in hot-wet conditions was considered in Part III of the review series. There are some additional points specific to fatigue testing under such conditions, to be made.

Several authors reported problems with grip tab failures. Rotem[246] used graphite cloth T300/5208 tabs after tabs manufactured from other materials (glass epoxy and aluminium) failed. Ryder and Walker[251], after some research, chose American Cyanimide FM400 as the best tab adhesive for testing at 82°C, 95% RH. For tension-compression tests, a temperature rise in the tab area of 39°C was recorded and resulted in tab failure. This problem was alleviated by cooling the tabs with RT air. In the gauge length, the temperature rise was 3°-8°C which was accounted for in the analysis of the results. Kan and Ratwani[161] in an earlier mentioned tests found that initial test frequency of 10 Hz resulted in a temperature rise of 2.8°C in the gauge area. Therefore, 5 Hz was chosen as the final loading frequency.

Some authors reported on spectrum loading tests with the environmental conditions simulated,[108, 230, 267] and include a brief description of the equipment which they have used to produce required temperatures and humidities around the test specimen. The thermal spike test in[108] for example, required equipment capable of achieving slew rates of 60°C/min. The cost of such equipment is very high and lower slew rates (~20°C/min) are more typical of such testing equipment. These high rates are necessary to conduct accelerated tests.

2.4 Conclusions

1. The large scatter apparent in composite tests is usually increased by varying test temperature and moisture conditions which makes interpretation of results difficult.

2. Non-organic fiber dominated laminates generally show little sensitivity to environmental factors under fatigue.

3. For matrix dominated laminates, fatigue characteristics are affected by temperature, while moisture, at room temperature, seems to have no effect. (This may vary greatly depending on the matrix used.)

4. Environmental effects on the viscoelastic properties of a matrix may have to be taken into account in composite fatigue analysis.

5. The observation of sensitivity or insensitivity to environmental factors is very closely tied to the definition of failure in composite materials.

6. Elevated temperature wet conditions and particularly environmental cycling reduce the fatigue resistance of composites. The slopes of S-N curves for graphite composites are relatively flat and even slight shifts of the curve will result in significant reductions in fatigue life. Strength reduction, especially in tension, may not be as significant.

7. Fatigue damage in composites usually is not directly related to residual strength. This is frequently ignored in fatigue studies of composites.

8. A method has been proposed for calculating fatigue properties of laminates, at various temperatures, from simple unidirectional and crossply studies at some reference temperature. This method is, however, in the very early stages of development.

9. The following deficiencies exist with respect to composite fatigue: a) A general theory for predicting laminate fatigue properties analogous to lamination theory for static properties; b) A general cumulative damage theory like Miner's rule for metals; c) A theory accounting for degradation of properties due to environmental factors. As a result, verification of existing designs has to be through testing under representative loads and environments.

10. Simple environmental simulation in accelerated tests should be adequate for fiber dominated materials while realistic environmental simulation is required for matrix dominated materials.

3.0 TIME DEPENDENT PROPERTIES

3.1 Mechanical Relaxation

Mechanical relaxation phenomena are observed when material behavior is nonelastic and stress and strain are not only functions of one another but also of time. The most commonly studied transient effects are creep and stress relaxation. In simple creep, either the applied stress or load is held constant while an increase in strain with time is recorded. Stress relaxation is observed when the stress required to hold a specimen at constant deformation is gradually decreasing with time. Results of creep and stress relaxation tests are strongly affected not only by the stress levels used, but by the test temperature, and for the case of organic solids (matrix materials), the moisture content of the specimen.

3.1.1 Linear viscoelasticity — superposition principle

The constitutive equation for general time-dependent material behavior can be written as follows:

$$\epsilon_z = f(\sigma_z, t, T, M, \sigma_H, T_H, M_H) \tag{1}$$

where:

ϵ_z — strain
σ_z — stress
t — time
T — temperature
M — moisture content
H — history of (temperature, moisture, stress)

This equation is so complex that it has never been used and instead, material behavior is approximated by combinations of elastic and viscous models.

The most general form of linear viscoelastic stress-strain relation in contracted engineering notation is given by[74]:

$$\sigma_i(t) = \int_{0^+}^{t} Q_{ij}(t-\tau) \frac{d\epsilon_j(\tau)}{d\tau} \, d\tau \tag{2}$$

Q_{ij} are the viscoelastic relaxation moduli. If conditions of temperature and moisture are varying, then the relaxation moduli become functions of these as well as time. In this case, strain in Equation (2) should be represented as sum of strains due to load, thermal and moisture expansion:

$$\epsilon_j(\tau) = \epsilon_j^*(\tau) - \alpha_j \Delta T(\tau) - \beta_j \Delta M(\tau) \tag{3}$$

α_j and β_j are coefficients of thermal and moisture expansion ΔT and ΔM are variations in temperature and moisture.

An alternate form of viscoelastic stress-strain relation which is more useful for creep type experiments is[33]:

$$\epsilon_i(t) = \int_{o^+}^{t} S_{ij}(t-\tau) \frac{d\sigma_j(\tau)}{d\tau} \, d\tau \tag{4}$$

where S_{ij} are the creep compliances.

The concept of superposition of time-temperature or time-temperature/moisture permits the use of master creep compliance (or relaxation modulus) curve representation of data:

$$S_{ij}(T, M, t) = S_{ij}(T_o, M_o, \zeta) \tag{5}$$

where T_o, M_o are reference temperature and moisture conditions and ζ is reduced time:

$$\zeta = \int_{o}^{t} \frac{d\eta}{a_{TM}(T, M)} \tag{6}$$

a_{TM} are horizontal shifting factors representing the amount of shifting necessary to bring the $S_{ij}(T, M, t)$ ($Q_{ij}(T, M, t)$) data into coincidence with the master curve. The relaxation tests (creep and stress relaxation) are usually conducted at constant conditions so Equation (6) becomes:

$$\zeta = \frac{t}{a_{TM}} \tag{7}$$

or taking logarithm of both sides of Equation (7)

$$\log_{10}\zeta = \log_{10} t - \log_{10} a_{TM} \tag{8}$$

Plots of isothermal/moisture moduli, or compliances, can be shifted horizontally with the magnitude of the shift equal to $\log_{10} a_{TM}$.

Materials which lend themselves to this type of operation are called thermo-rheologically simple (TSM). Vertical shifting is required when horizontal shifting did not result in a smooth, well-defined master curve. The material is then termed thermo-rheologically complex (TCM).

For rigid plastics with and without reinforcement, the creep compliance relationship frequently used for approximation is:

$$S(t) = S_o + S_1\, t^n \tag{9}$$

S_o, S_1, n — constant with time.

For TSM, the creep compliance in a series of isothermal tests is given by:

$$S = S_o + S_1 \left(\frac{t}{a_T}\right)^n \tag{10}$$

For TCM S_o is temperature dependent:

$$S = S_o(T) + S_1 \left(\frac{t}{a_T}\right)^n \tag{11}$$

The linear viscoelastic model of time-dependent material behavior is used because of its simplicity. The superposition principle, when applicable, dramatically reduces the amount of time and number of data points required to fully characterize the viscoelastic properties of the material. However, composite materials generally do not follow this approximation so it can be used only for limited conditions (usually low stress and temperature levels) on an individual basis[33, 328].

3.1.2 Time-temperature superposition

Yeow, Morris and Brinson[328] studied time-temperature behavior of unidirectional graphite/epoxy (T300/934). The stress levels used in this study were such that the applied axial stress did not exceed 10% UTS. Specimens were subjected to various loads (mechanically conditioned) to determine whether load history affected material response. Since the stress-strain curves did not change, the number of specimens required for testing was reduced (specimens could be used several times). The material was tested for linearity — creep and recovery for [90°] specimen must have equal instantaneous strain (ϵ_o in Fig. 20) and the stress-strain curve after 15 minutes of creep must be linear (Fig. 21). Isothermal creep tests were conducted for temperatures of 20°C to 210°C and results for the reciprocal of reduced compliance ($1/S_{22}$) are shown in Figure 22. Reduced compliance is defined as:

$$S_{22} = \frac{\epsilon_2(t)}{\sigma_o}\;\frac{T}{T_o} \tag{12}$$

where

σ_o — applied stress

T_o — reference temperature

T — temperature of the test

$\epsilon_2(t)$ — strain (transverse to fiber direction)

The master curve obtained from these data is shown in Figure 23 while shift factors a_T are plotted in Figure 24. It was found that shift factors for all fiber orientations as well as for the reduced shear modulus S_{66}, are equal. The deviation of the shift factor for reduced Poisson coefficient ν_{21} was attributed to data scatter.

Fiber dominated properties (S_{11} and ν_{12}) were found to be time and temperature insensitive.

Beckwith[33] reported on a study of Shell 58-68R epoxy and its composite with S-901 glass fiber. The temperature range used in the study was −7°C to 60°C. Tests were single and multiple (3) cycle creep and recovery. Both the resin and composite were found to be TCM materials as initial compliance (S_0 see Eq. (11)) was temperature dependent (Figs. 25 and 26). The master curve for net creep compliance was obtained for the epoxy after initial compliance for each temperature was subtracted from the total compliance measured (Fig. 27). The glass/epoxy tested can be regarded as linearly viscoelastic. The range of linearity depends on temperature and fiber angle. At 24°C (±45°) glass/epoxy recovers completely for 35 MPa but not for higher loads. However at 60°C it does not recover even for 20 MPa (Fig. 28). Beckwith observed a change in initial and net creep compliance in composites due to multiple loading. The nonlinear effects were assumed to be predominantly microcracking and the crack growth seemed to cause a disproportionate amount of damage during the first few loading cycles.

For those cases where the superposition principle can be applied, the key to obtaining the master curve are the time-temperature shift factors. Kibler and Carter[167] examined the possibility of using electrical conductivity measurements for obtaining these factors.

Direct-current resistance of neat epoxies is very high. It can be therefore assumed that charge transport proceeds by the short range migration of heavy ions. In that case, the direct-current electrical conductivity is itself inversely proportional to viscosity. Samples of 5208 epoxy resin were used to extract the time-temperature shift factors from thermomechanical and electrical conductivity measurements:

$$a_T = \frac{\eta(T)}{\eta(T_R)} = \frac{\sigma(T_R)}{\sigma(T)} \tag{13}$$

where η - viscosity σ - electrical conductivity. Only linear effects were studied. The a_T factors extracted from both methods are compared in Figure 29. Carter and Kibler concluded that conductivity data may be useful in obtaining quick estimates of the effects of temperature on time-dependent mechanical response.

3.1.3 Time-temperature-moisture superposition

The considerable 'plasticizing' effect of moisture on matrix type resins relaxation modulus can be seen in Figure 30 taken from Browning[42, 44]. The two master curves shown correspond to dry and wet (equilibrium wt. gain at 71°C, 100% RH) samples. A shift factor from wet to dry was found to be 10. This implies that the same response will be exhibited by a wet sample 10 times faster then by the dry sample.

The effect of moisture on time-dependent response in composites has been studied only recently and by relatively few authors. In one of the earlier works Wang and Liu[304] studied creep in graphite/epoxy (Modmor II/1004) unidirectional composite. Specimens were placed in various humidity conditions at 21°C and 65°C. A significant increase in creep with humidity and temperature was observed (Figs. 31 and 32). Within the limits of linear response, the time-temperature-humidity shifting was shown to be effective (Fig. 33).

A much broader study was undertaken by Crossman et al[70]. The materials used were GY70/CE339, T300/5209 (both unidirectional) and HMF 330C/934 (T300 satin weave). All these materials are graphite/epoxies. Specimens were exposed to humidity conditions until saturation prior to stress relaxation testing. Results are shown in Figures 34, 35 and 36. For HMF 330C/934, the relaxation modulus at 149°C dry corresponds to the 71°C wet values. Part of this study was directed at demonstrating the effect of relaxation effects on residual stresses (see Part II of this review series).

Kibler[168] presented creep test results for T300/5208 and AS-3502 graphite/epoxies. Only the linear range of behavior was studied (stress levels at 25% — 35% UTS). The creep compliance master curve (Fig. 37) is essentially the same for both materials. Longer-term tests agree well with short-term tests. For fiber-dominated properties no time dependence was found (Fig. 38). From the above data G_R (t) was calculated and is shown in Figure 39. It can be seen that when both moisture and elevated temperature are present, the shifts are considerable.

3.1.4 Nonlinear responses and time-temperature-stress superposition

Composite materials generally demonstrate nonlinear time-dependent behavior. Linear viscoelasticity is applied only for low stresses. Wang and Liu[304] encountered nonlinear response at 65°C, 95% RH at a stress level of 29% UTS (Fig. 32). This stress level is rather low from a design point of view. Wang and Wang[301] measured creep response at glass/epoxy (Scotchply 1002) at various load levels with controlled temperature and moisture content. For (90°) laminates the degradation of material is found to be quite serious when the temperature and moisture content increase (Figs. 40 and 41). Similar behavior was found for (±45°) specimens. Even the (0°) laminate, at high temperature and moisture exhibited a definite increase in creep strain (Fig. 42).

Griffith, Morris and Brinson[114] demonstrated that in some cases the time-temperature-stress superposition principle (TTSSP) can be used to produce unified master curves reduced to particular stress-temperature conditions. The procedure is explained schematically in Figure 43. The method is regarded to be valid if smooth curves can be produced. For the T300/934 material tested good correlation was obtained between the master curve from short-term tests and a long-term test (Fig. 44).

3.1.5 Predictions

a) Micromechanics — Halpin-Tsai

Halpin-Tsai micromechanics equation was used[168, 73] to predict viscoelastic modulus from bulk properties with good correlations (Fig. 45).

b) Macromechanics

Various authors have adopted elastic lamination theory to account for time-dependent behavior[168, 328, 114, 301, 74].

As a first step, it is necessary to calculate the transformed reduced compliances for a lamina arbitrarily oriented with respect to a laminate or global axis system. Yeow et al[328] used a visco-elastic analog of elastic orthotropic equation and compared it with the master curve from short-term data and long-term data (Fig. 46). Correlation was good, however, in a more recent publication, Griffith et al[114] concluded that all axis predictions must account for the stress-dependent nature of the master curves. The uniaxial stress must be transformed into stress components in the principal material directions and master curves for stresses associated with these directions should then be used with the transformation equation. For this approach, prediction was within 10% of measured responses.

A procedure for calculating creep response of a laminate from measured creep responses of laminae was shown in[301] and the results of comparison of predictions with measurements are in Figure 47. This procedure allows for the nonlinear nature of creep responses. For linear viscoelastic approximations, computer programs based on an elastic response model and data from master curves were used with satisfactory results[168].

3.2 Stress-Rupture

Creep tests can be carried out to failure (stress-rupture). For this case, the life of a composite does not seem to correlate to the initial static strength or to the residual static strength taken at any point before rupture. More experimental data is needed to determine times to rupture characteristics. As several years may be needed before rupture occurs accelerated testing methods are required. Chiao et al[60] and later Hahn and Chiao[120] compared long-term test results to results from a time-temperature reduction based on an Arrhenius type of equation. The materials tested were Kevlar 49 and S-glass strands impregnated with epoxy resins (several resins were used). In the earlier work[60] a discrepancy was found between the predicted and measured rupture times for stresses over 85% UTS. In[120] lifetime data spanning over eight years have been analyzed by a two-parameter Weibull distribution and it was found that above 80% UTS the failure process changes from a wear-out type to initial defect controlled. At high temperatures the failure process was also wear-out type and this explained the difference between the predicted and experimental data for higher stresses. The stress rupture of S-glass epoxy composite was found to be a random failure process (shape parameter of Weibull distribution $\alpha \approx 1$) regardless of stress level. The logarithmic characteristic lifetime was linearly related to the applied stresses.

Aveston et al[26] tested fiber bundles unimpregnated and impregnated with resins in wet environments. For carbon fibers no effect of time was observed. For E-glass considerable degradation was observed with worst effects of immersion in water of epoxy and polyester impregnated strands. Cemfil — glass fiber (alkali resistant) demonstrated much better performance while Kevlar 49 performance was somewhere between E-glass and Cemfil.

Allen[8] suggested the use of flexural creep and rupture tests to screen reinforced epoxy resins for environmental performance. Creep rates and rupture incidence are increased by immersion of specimens in water.

3.3 Conclusions

1. The linear viscoelastic model is a useful approximation for predicting CM performance at low stress levels and moderate conditions of temperature and moisture.

2. Fiber dominated properties (compliance S_{11} and Poisson ratio) are time and temperature/moisture insensitive (graphite) or very slightly sensitive (glass and Kevlar 49).

3. For pressure vessels and similar applications where material remains loaded for extensive periods, stress rupture in the fiber direction is a possibility. Arrhenius type relations may be used to predict time to rupture from short-term data.

4. Most composites are thermo-rheologically complex materials as initial compliance depends on testing conditions.

5. Moisture has a considerable "plasticizing" effect and the same creep response may be expected from a wet sample 10 times sooner then from a dry sample.

6. The Halpin-Tsai equation predicts the viscoelastic modulus from bulk properties with good results.

7. Lamination theory may be adopted to successfully predict the laminate viscoelastic response, both linear and nonlinear.

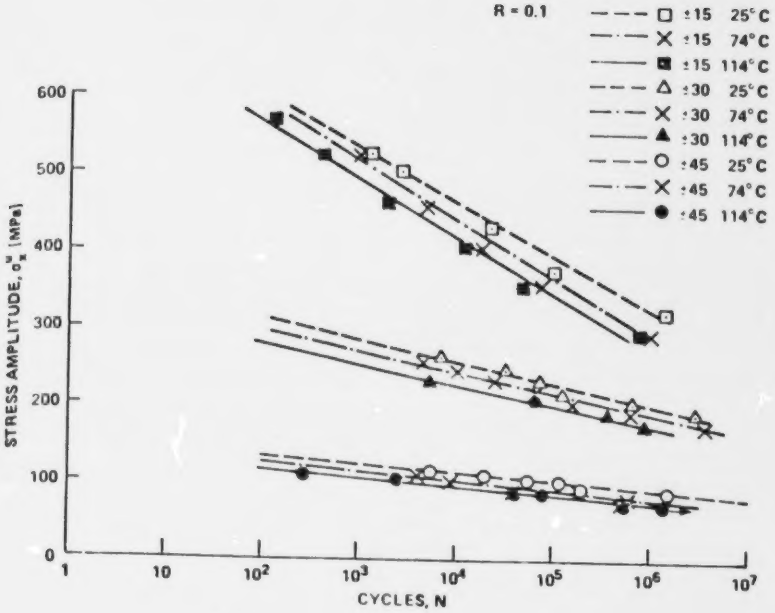


FIG. 1: S-N CURVES OF SOME ANGLE-PLY LAMINATES[245]

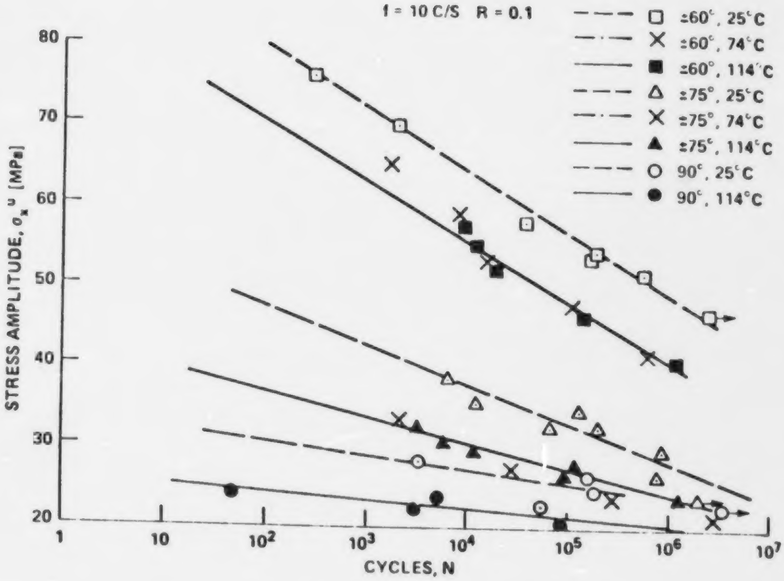


FIG. 2: S-N CURVES OF SOME ANGLE-PLY LAMINATES[245]

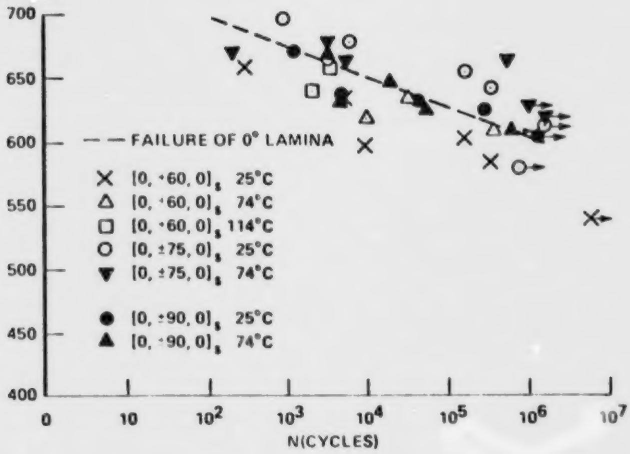


FIG. 3: S-N CURVES OF SOME SYMMETRICALLY BALANCED LAMINATES THAT FAIL BY FIBER FRACTURE[245]

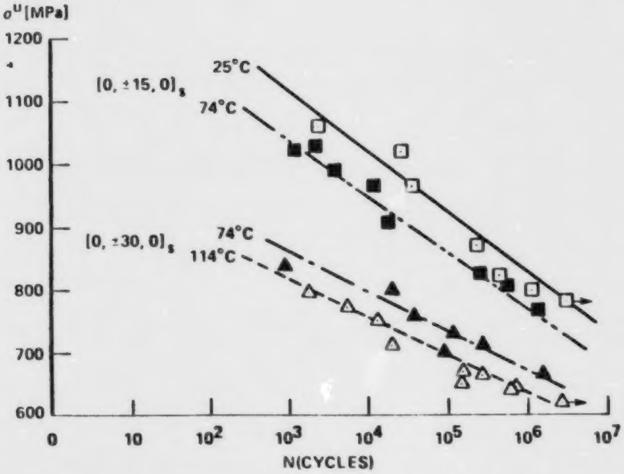


FIG. 4: S-N CURVES OF SOME SYMMETRICALLY BALANCED LAMINATES, SHIFTED WITH TEMPERATURE[245]

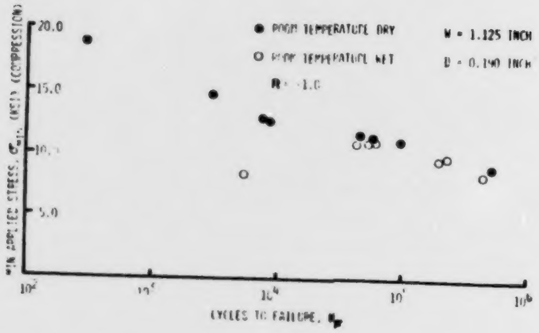


FIG. 5: S-N CURVE FOR LAMINATE 1 SPECIMENS [RTD AND RTW][161]

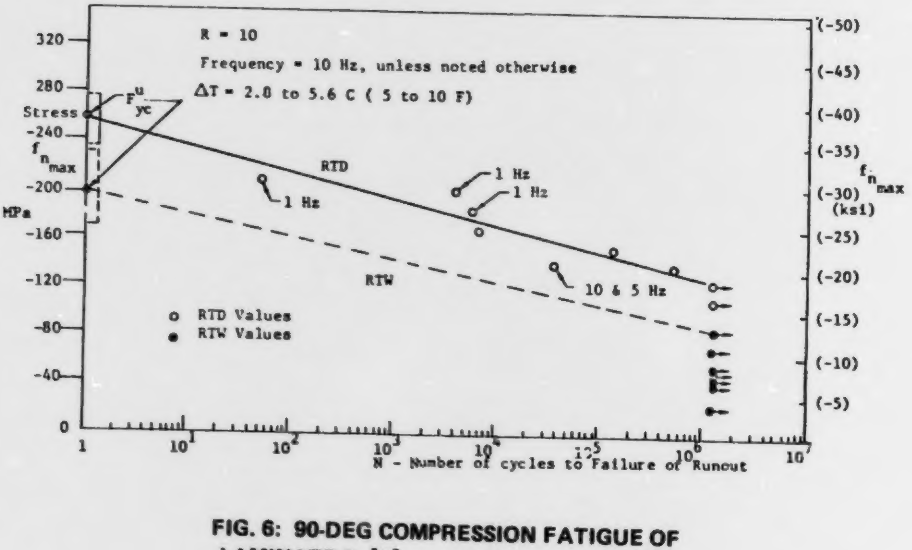
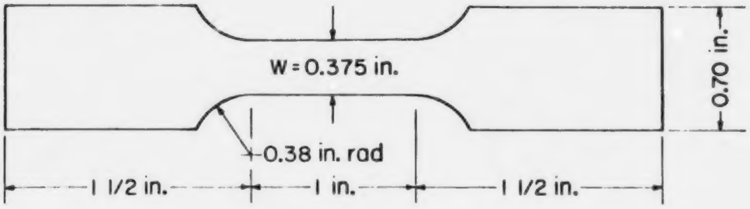
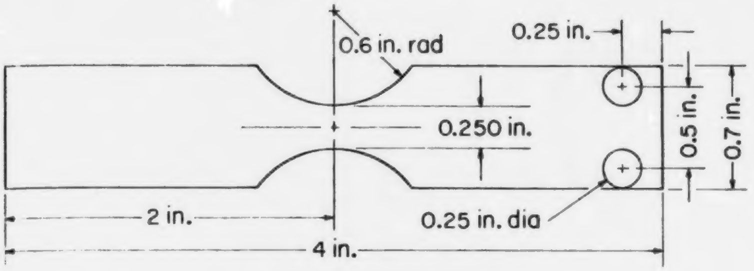


FIG. 6: 90-DEG COMPRESSION FATIGUE OF LAMINATE B, $[0]_{24T}$ – RTD AND RTW[115]


W = 0.375 in.
0.70 in.
0.38 in. rad
1 1/2 in.
1 in.
1 1/2 in.

(a) TORSION SPECIMEN


0.6 in. rad
0.25 in.
0.250 in.
0.5 in.
0.7 in.
2 in.
0.25 in. dia
4 in.

(b) FATIGUE SPECIMEN

FIG. 7: TEST SPECIMENS[277]

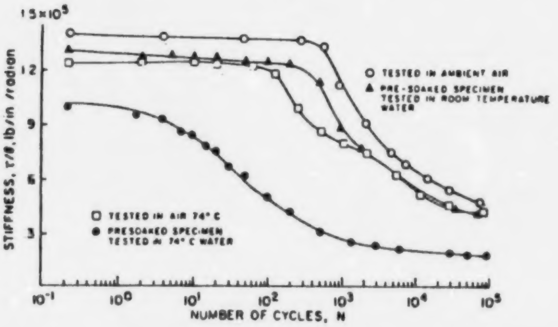


FIG. 8: TORSION FATIGUE OF ±45° FIBER-ORIENTED GRAPHITE-EPOXY COMPOSITE. DECREASE IN STIFFNESS [τ/θ] WITH NUMBER OF CYCLES (LOG N) AS A FUNCTION OF ENVIRONMENT. ±45° FIBER ORIENTATION. $\Delta\tau_o$ = ±11,200. TEST FREQUENCY 1 Hz[278]

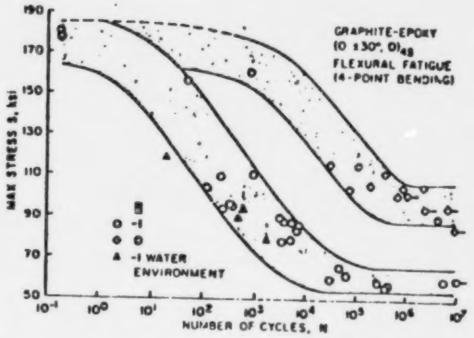


FIG. 9: FLEXURE FATIGUE TESTS OF ±30° FIBER-ORIENTED SPECIMENS IN REVERSE (R = −1) AND REPEATED (R = 0) CYCLING[278]

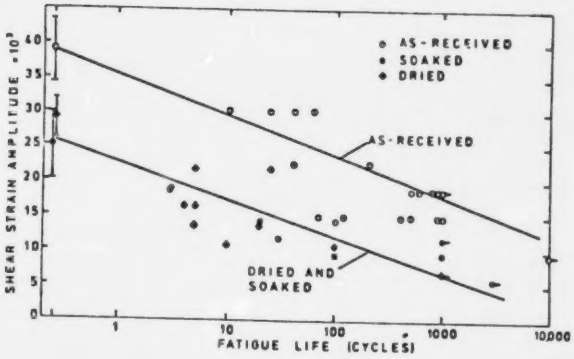


FIG. 10: FATIGUE LIVES OF CFRP UNDER CONSTANT SHEAR STRAIN AMPLITUDE CYCLING AFTER VARIOUS TREATMENTS[233]

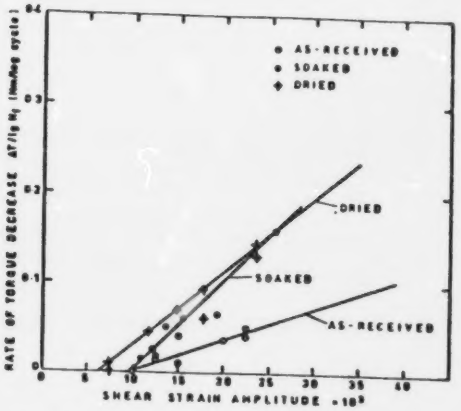


FIG. 11: ΔT/LOG N DURING CONSTANT STRAIN AMPLITUDE CYCLING OF KFRP[233]

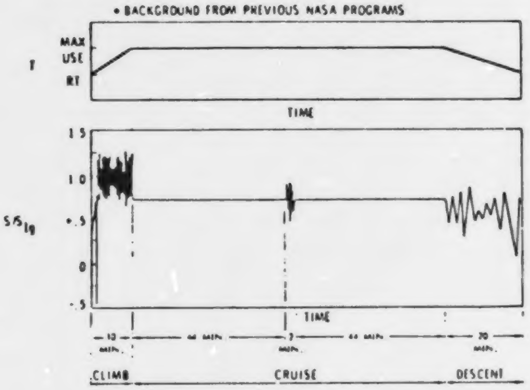


FIG. 12: LOAD/THERMAL HISTORY IS REALISTIC[126]

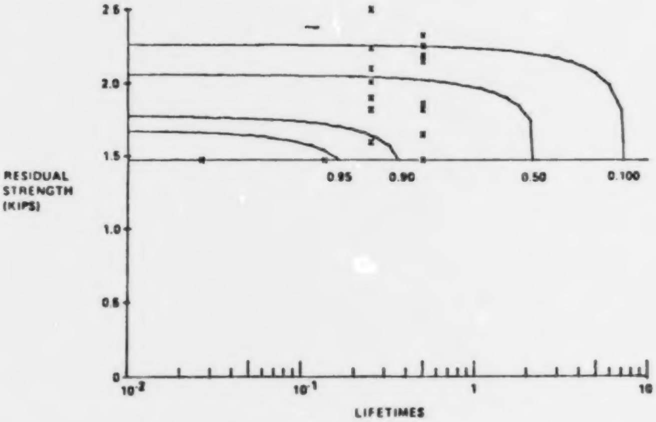


FIG. 13: WEAROUT MODEL FOR UNNOTCHED $[0/\pm 45]_s$ A-S/3501 GRAPHITE EPOXY[126]

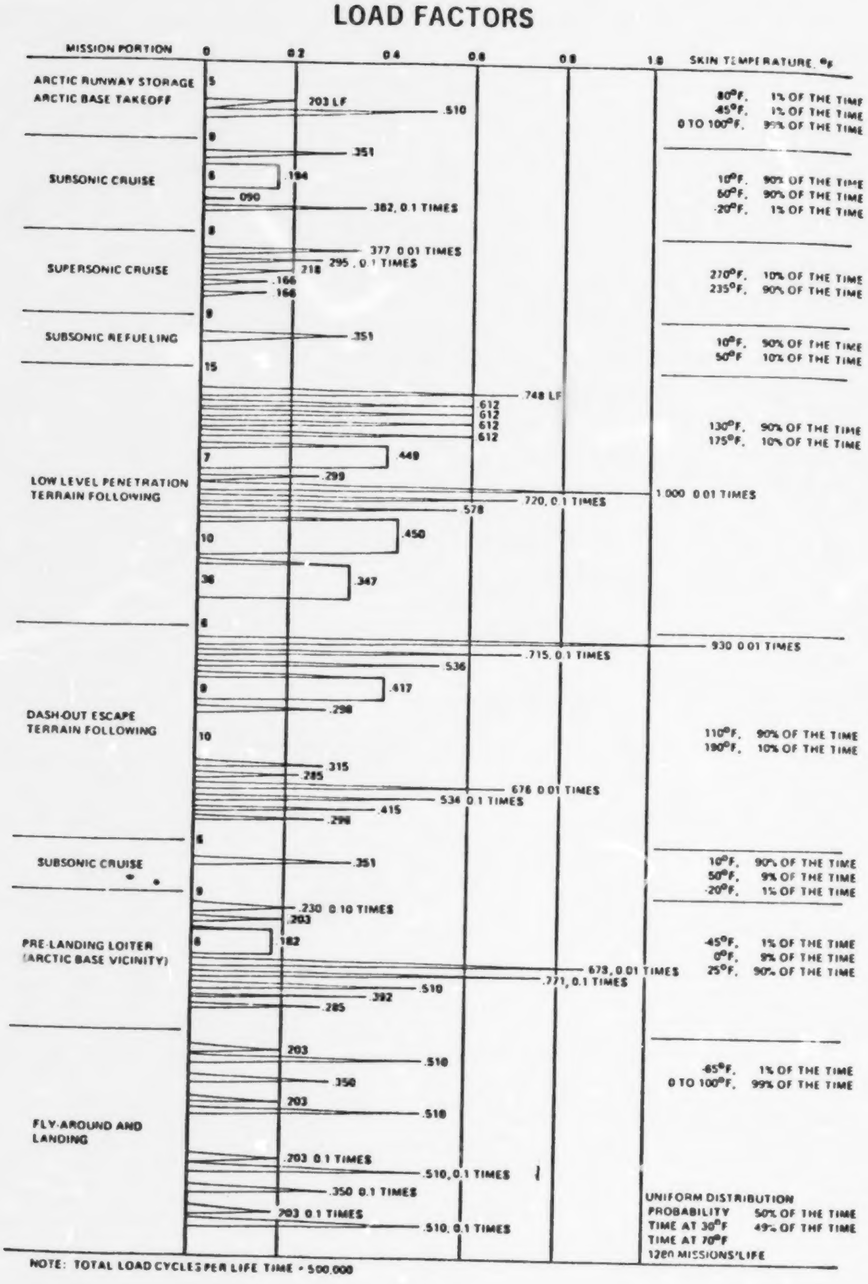


FIG. 14: SKIN TENSILE STRESS HISTORY[230]

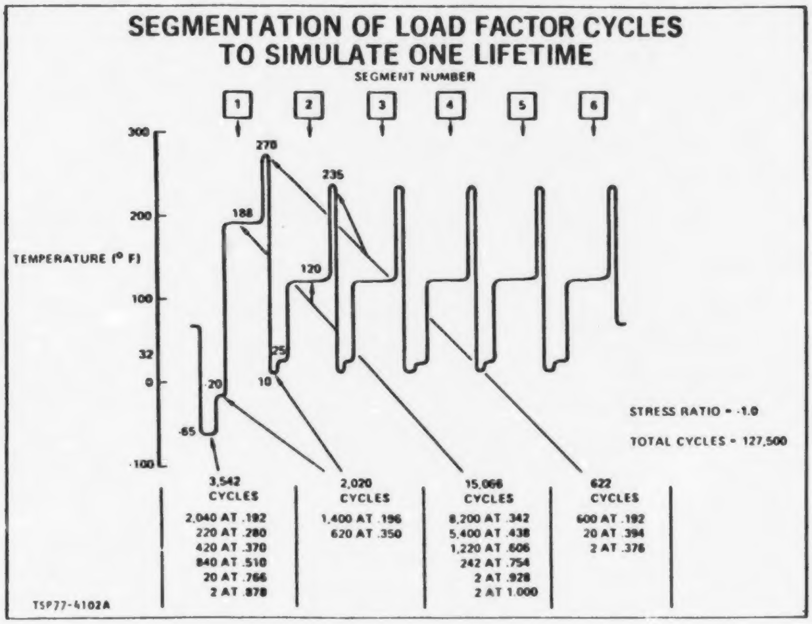
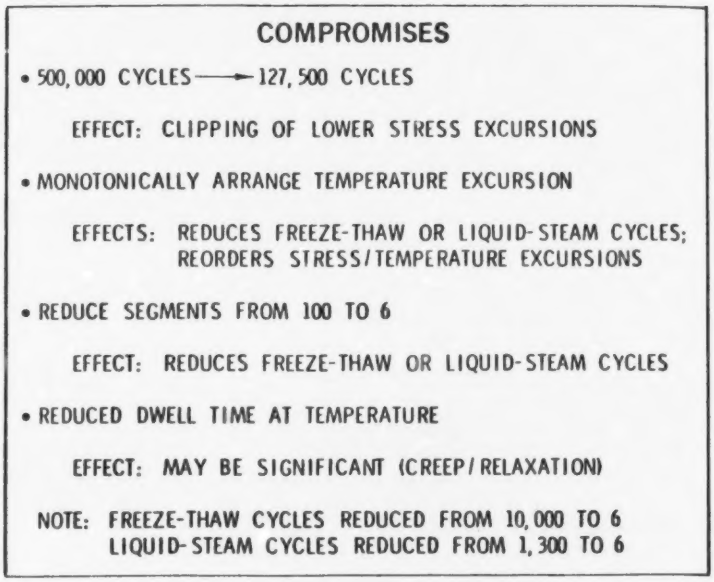


FIG. 15: SEGMENTATION OF LOAD/TEMPERATURE CYCLES[230]



COMPROMISES

- 500,000 CYCLES ——→ 127,500 CYCLES

  EFFECT: CLIPPING OF LOWER STRESS EXCURSIONS

- MONOTONICALLY ARRANGE TEMPERATURE EXCURSION

  EFFECTS: REDUCES FREEZE-THAW OR LIQUID-STEAM CYCLES;
  REORDERS STRESS/TEMPERATURE EXCURSIONS

- REDUCE SEGMENTS FROM 100 TO 6

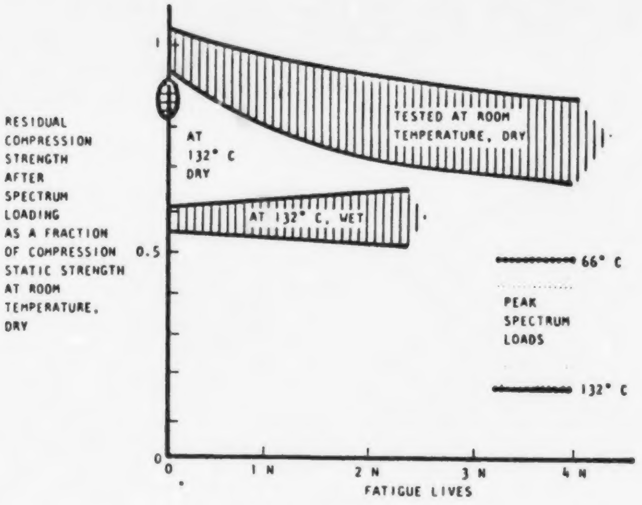
  EFFECT: REDUCES FREEZE-THAW OR LIQUID-STEAM CYCLES

- REDUCED DWELL TIME AT TEMPERATURE

  EFFECT: MAY BE SIGNIFICANT (CREEP/RELAXATION)

NOTE: FREEZE-THAW CYCLES REDUCED FROM 10,000 TO 6
LIQUID-STEAM CYCLES REDUCED FROM 1,300 TO 6

FIG. 16: COMPROMISES[230]



NOTE: 1 ON THE ORDINATE DESIGNATES 87,000 PSI)0.6 GP$_c$)

FIG. 17: RESIDUAL STRENGTH OF FATIGUE AS/3501-5-DEGRADES WITH TEMPERATURE, HYDRATION, PROOF LOADING, AND SIZE OF IMPERFECTION OR DAMAGE[176]

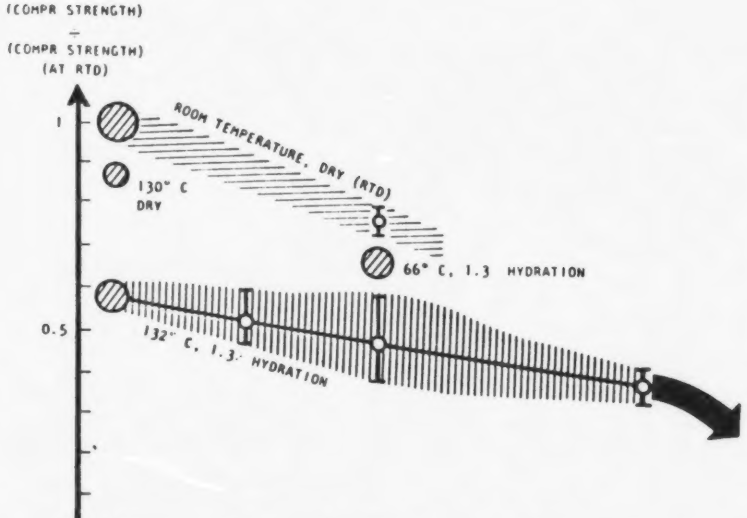


FIG. 18: THE COMPRESSIVE STRENGTH OF AS/3501-5-DEGRADES WITH TEMPERATURE, HYDRATION, PROOF LOADING, AND SIZE OF IMPERFECTION OR DAMAGE[176]

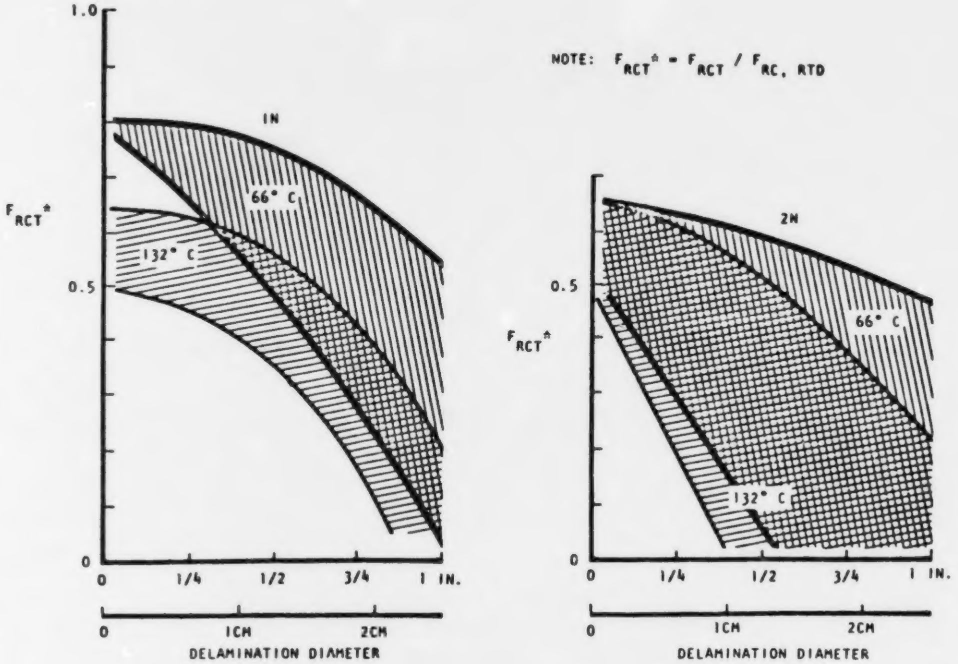


FIG. 19: RESIDUAL STRENGTH AT 66 AND 132°C (150 AND 270°F) OF FATIGUE GRAPHITE/EPOXY WITH DELAMINATIONS OF VARIOUS EXTENT[176]

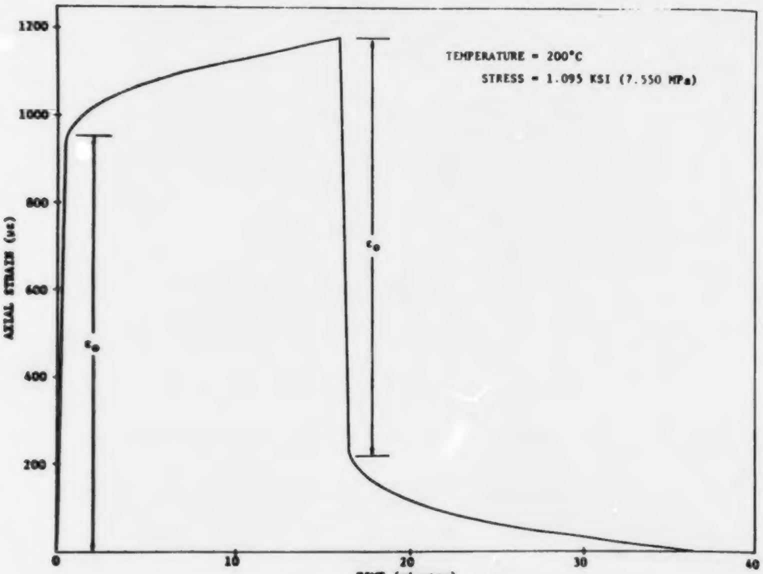


FIG. 20: CREEP AND CREEP RECOVERY OF $[90°]_{8s}$ LAMINATE[328]

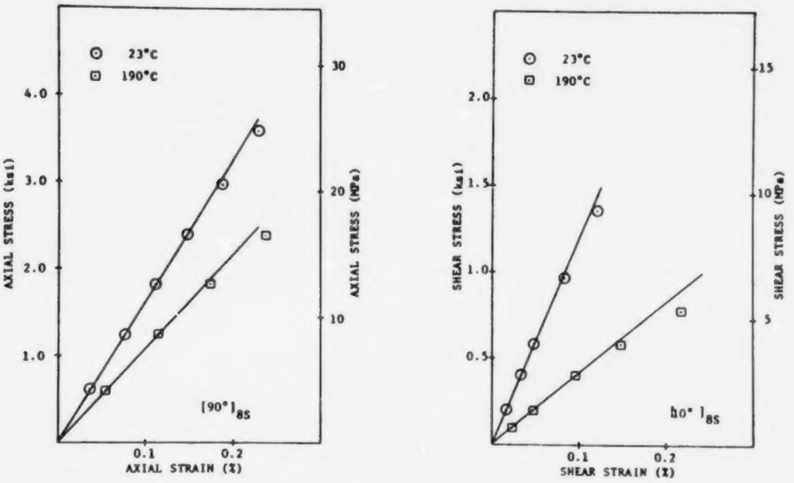


FIG. 21: LINEARITY CHECK (STRESS-STRAIN CURVES AFTER 15-MIN CREEP)[328]

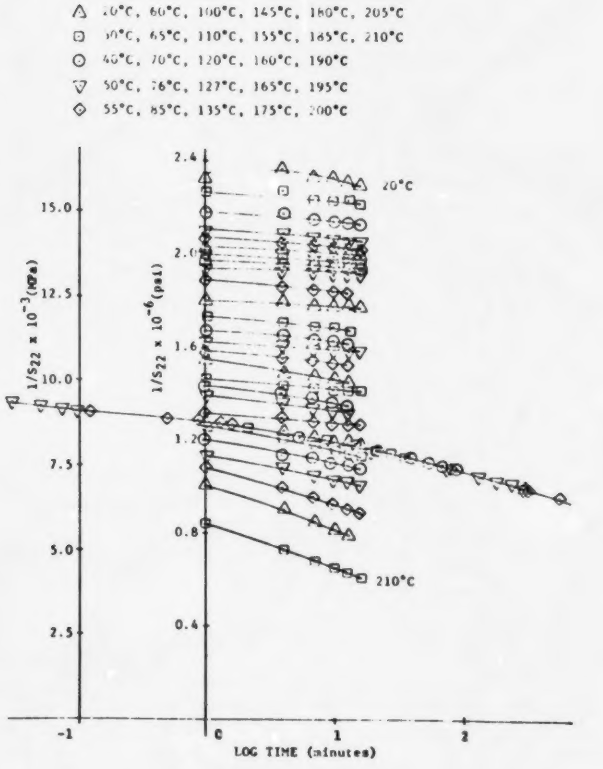


FIG. 22: REDUCED RECIPROCAL OF COMPLIANCE, $1/S_{22}$, AND PORTION OF 180°C MASTER CURVE FOR $[90^\circ]_{8s}$ T300/934 GRAPHITE/EPOXY LAMINATE[328]

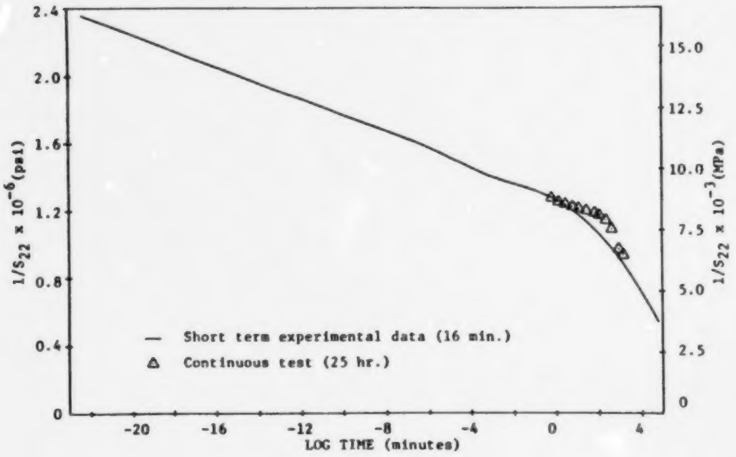


FIG. 23: MASTER CURVE OF THE RECIPROCAL OF REDUCED COMPLIANCE, $1/S_{22}$, OF $[90°]_{8s}$ LAMINATE AT 180°C[328]

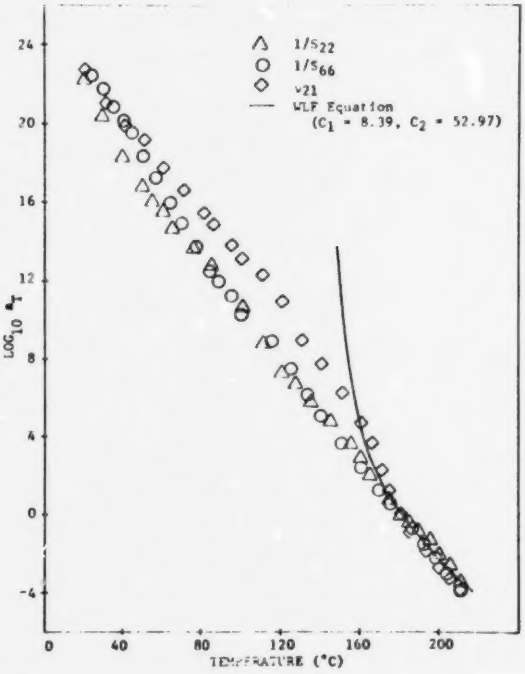


FIG. 24: LOG a_T VERSUS TEMPERATURE[328]

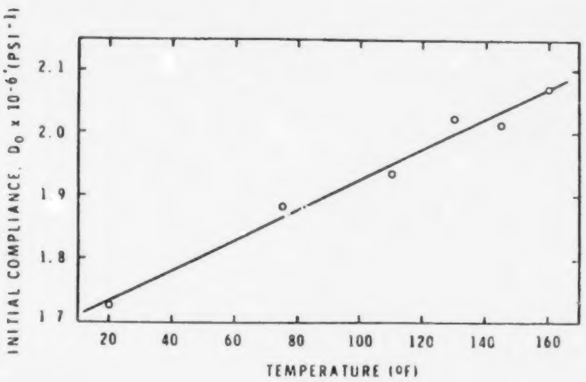


FIG. 25: TEMPERATURE DEPENDENCE OF INITIAL COMPLIANCE, D_o FOR SHELL 58-68R EPOXY[33]

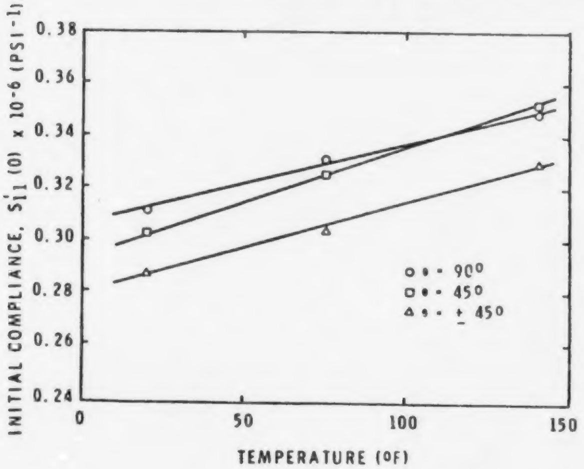


FIG. 26: TEMPERATURE DEPENDENCE OF INITITAL CREEP COMPLIANCES FOR THE GLASS/EPOXY COMPOSITE[33]

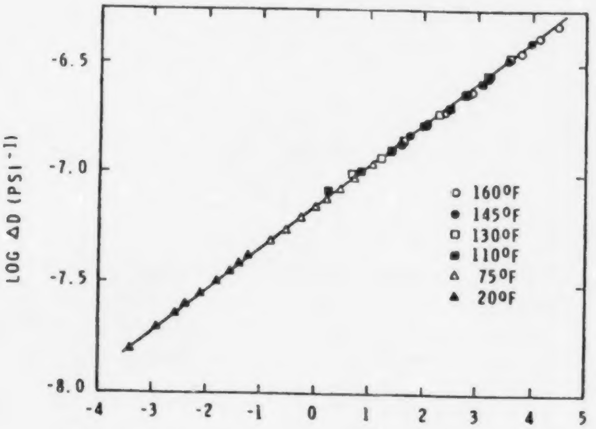


FIG. 27: MASTER CURVE FOR NET CREEP COMPLIANCE, ΔD, FOR SHELL 58-68R EPOXY[33]

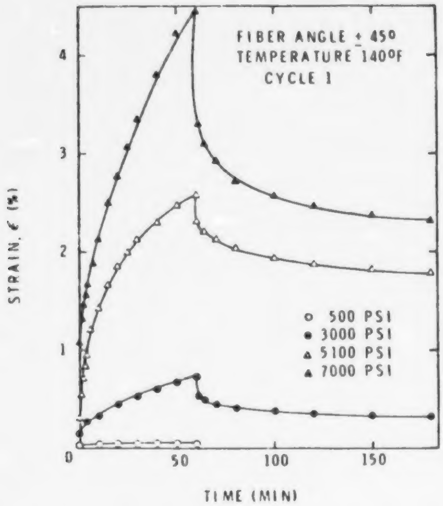


FIG. 28: CREEP AND RECOVERY ±45° GLASS/EPOXY AT 140°F (60°C)[33]

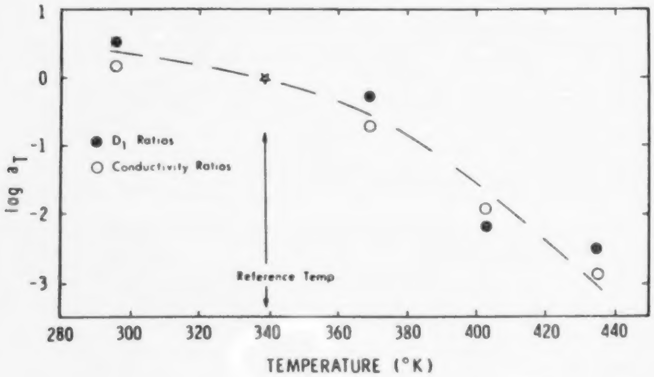


FIG. 29: TEMPERATURE DEPENDENCE OF THE SHIFT FACTOR, a_T, DETERMINED FROM ELECTRICAL CONDUCTIVITY MEASUREMENTS AND ISOTHERMAL CREEP COMPLIANCES[167]

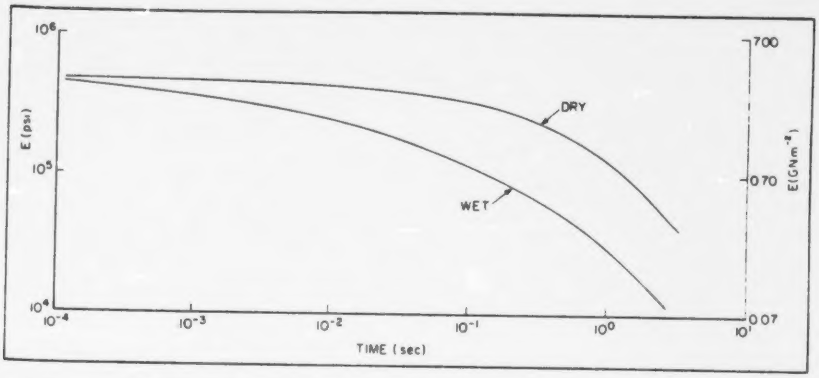


FIG. 30: RELAXATION MODULUS MASTER CURVES, REFERENCE TEMPERATURE = 300°F (149°C). EPOXY RESIN – TETRAGLYCIDYLMETHYLENE DIANILINE DIAMINE CURING AGENT[42]

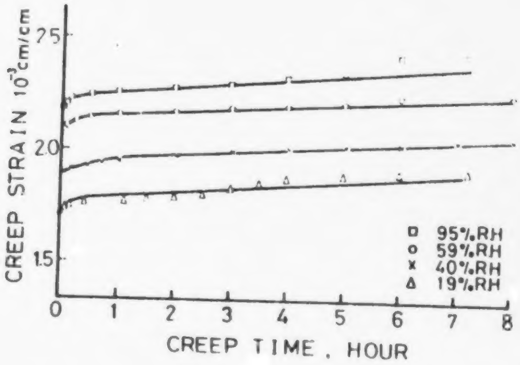


FIG. 31: CREEP STRAIN HISTORY – 21°C[304]

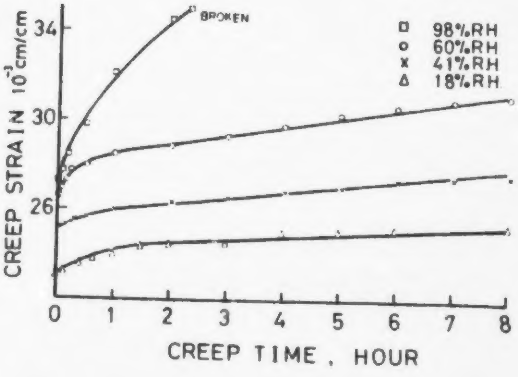


FIG. 32: CREEP STRAIN HISTORY – 65°C[304]

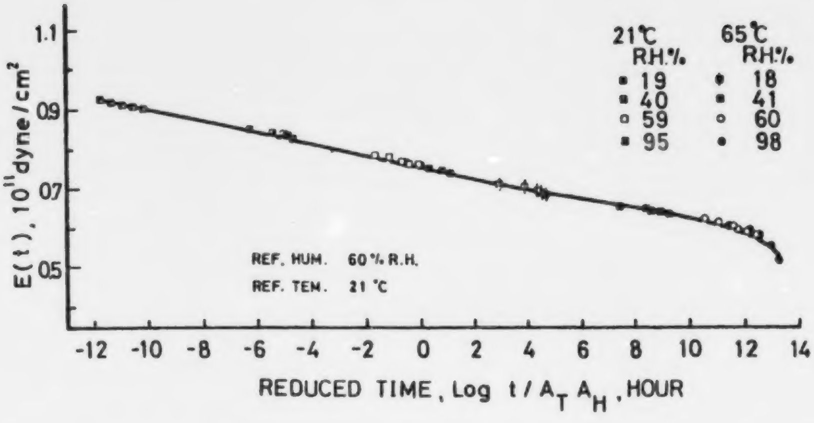
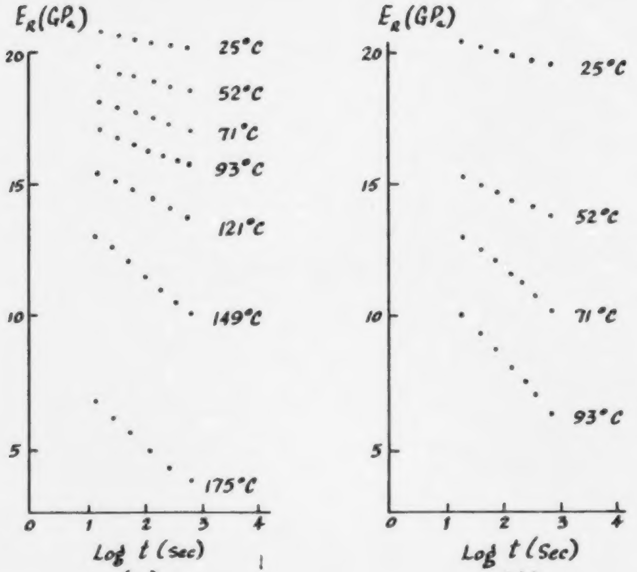


FIG. 33: COMPOSITE (MASTER) RELAXATION MODULUS UNDER TEMPERATURE-HUMIDITY EFFECTS[304]



(a)

(b)

FIG. 34: RELAXATION MODULUS OF $(\pm 45)_{2s}$ T300/934 LAMINATES CONTAINING (a) 0.14 PERCENT MOISTURE AND (b) 1.40 PERCENT MOISTURE[70]

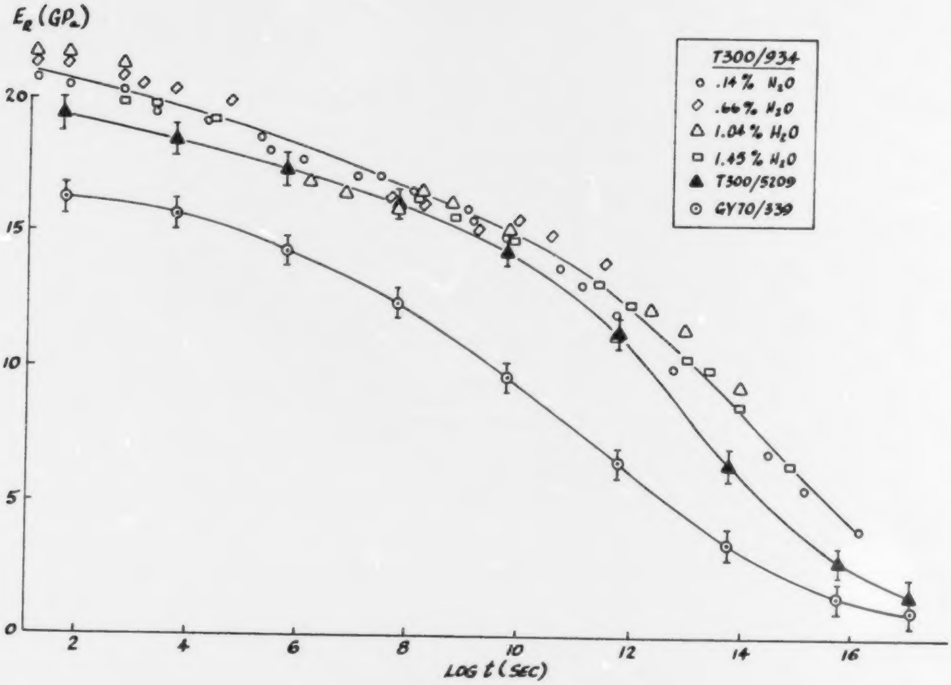


FIG. 35: MASTER RELAXATION MODULUS CURVES FOR $(\pm 45)_{2s}$ LAMINATES OF THREE COMPOSITE MATERIALS. HORIZONTALLY SHIFTED T300/934 DATA ARE SHOWN[70]

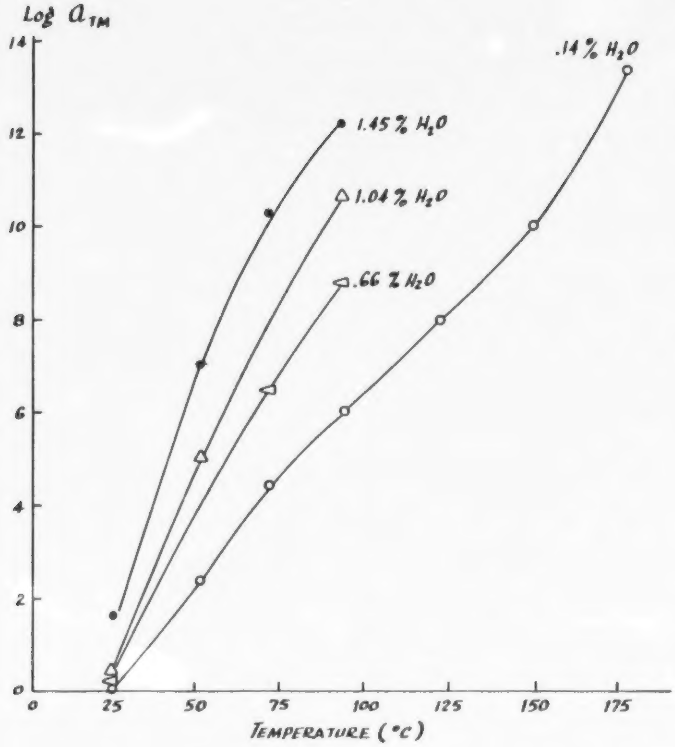


FIG. 36: SHIFT FACTOR a_{TM} FOR T300/934 VERSUS TEMPERATURE AND MOISTURE[70]

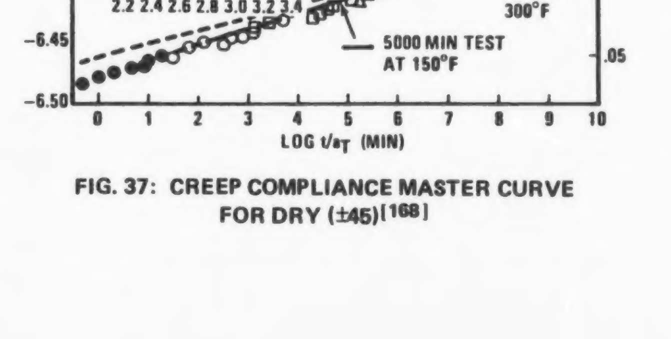


FIG. 37: CREEP COMPLIANCE MASTER CURVE FOR DRY (±45)[168]

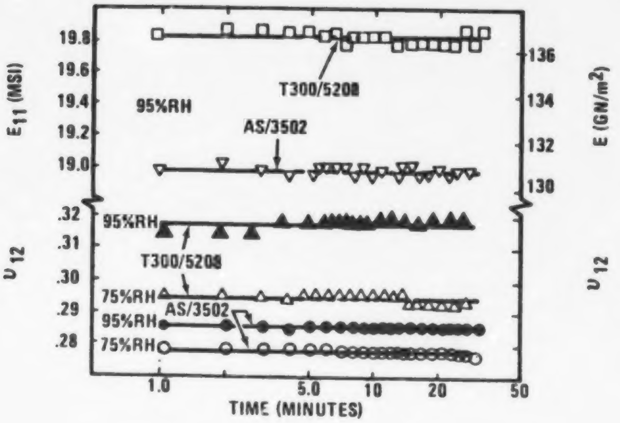


FIG. 38: TYPICAL E_{11} AND ν_{12} FOR RT WET $(0)_6$ COUPONS[168]

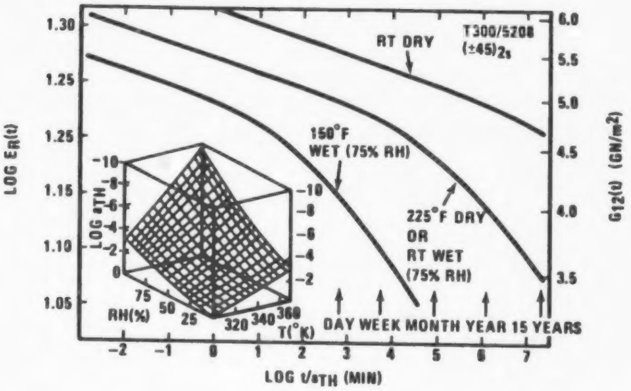


FIG. 39: RELAXATION MODULI FOR DRY (±45)[168]

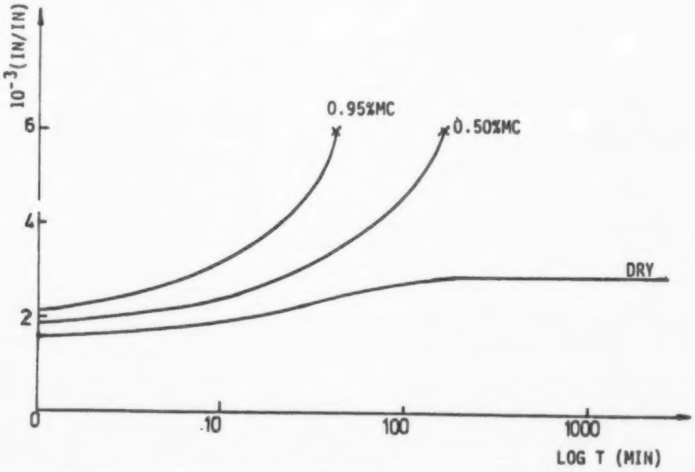


FIG. 40: CREEP OF (90°) LAMINATE AT 215°F (102°C) AND VARIOUS MOISTURE CONTENTS (900 psi = 6.2 MPa)[301]

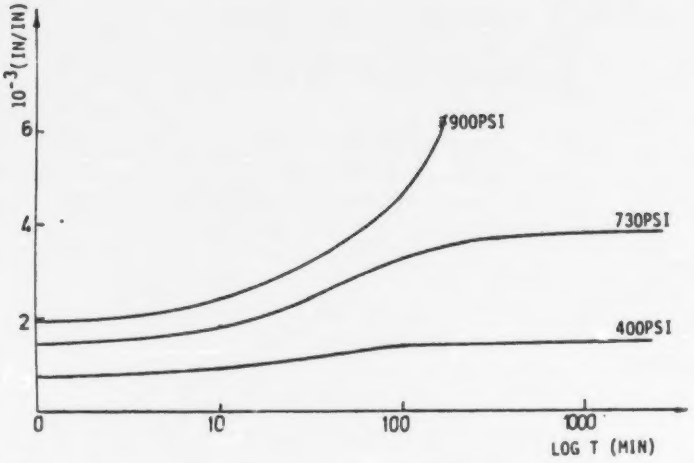


FIG. 41: THE EFFECT OF STRESS LEVEL ON CREEP STRAIN FOR (90°) LAMINATES AT 215°F (102°C) AND 0.5% MC[301]

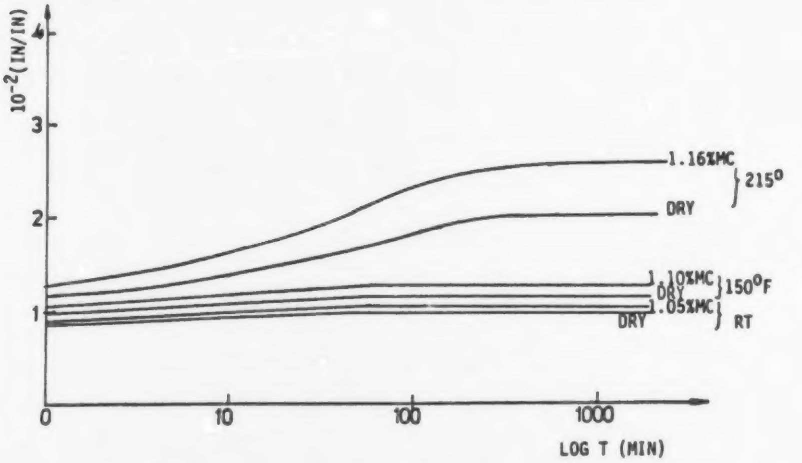
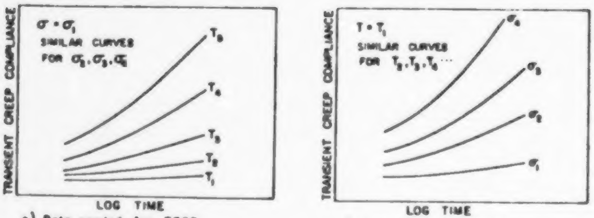
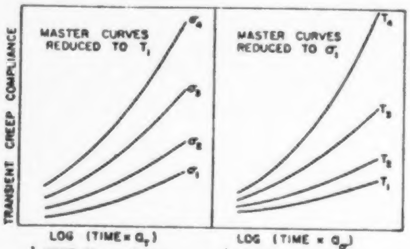


FIG. 42: CREEP OF (0°) LAMINATE UNDER VARIOUS ENVIRONMENTAL CONDITION (46600 psi = 321 MPa)[301]


TRANSIENT CREEP COMPLIANCE
$\sigma = \sigma_1$
SIMILAR CURVES
FOR $\sigma_2, \sigma_3, \sigma_4$
T_4
T_3
T_2
T_1
LOG TIME
$T = T_1$
SIMILAR CURVES
FOR $T_2, T_3, T_4 \cdots$
σ_4
σ_3
σ_2
σ_1

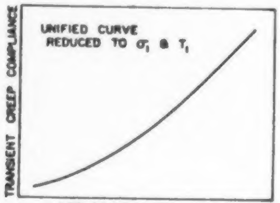
a) Data needed for TTSP

b) Data needed for TSSP


TRANSIENT CREEP COMPLIANCE
MASTER CURVES
REDUCED TO T_1
σ_4
σ_3
σ_2
σ_1
LOG (TIME × a_T)
MASTER CURVES
REDUCED TO σ_1
T_4
T_3
T_2
T_1
LOG (TIME × a_σ)

c) TTSP Master Curves

d) TSSP Master Curves


TRANSIENT CREEP COMPLIANCE
UNIFIED CURVE
REDUCED TO σ_1 & T_1
LOG (TIME × a_T × a_σ)

e) TSTSP Master Curve

FIG. 43: SCHEMATIC DIAGRAM TO ILLUSTRATE THE TIME-STRESS-TEMPERATURE SUPERPOSITION PRINCIPLE[114]

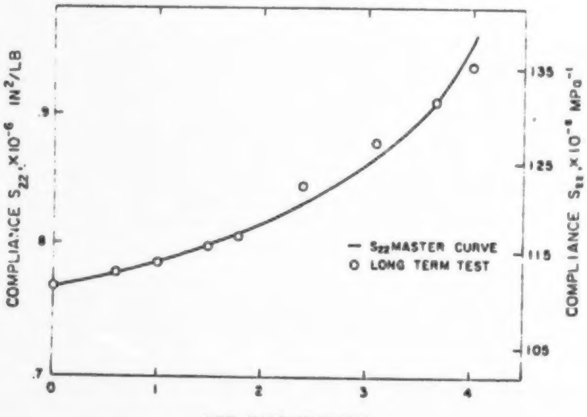


FIG. 44: COMPARISON OF S_{22} MASTER CURVE WITH A LONG TERM TEST AT 320°F (160°C) AND 2,750 psi (19 MPa)[114]

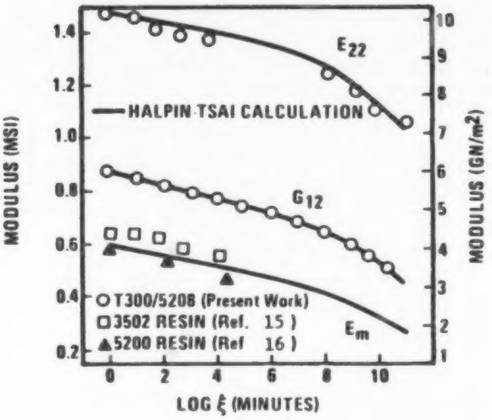


FIG. 45: MEASURED AND PREDICTED LAMINATE PROPERTIES[168]

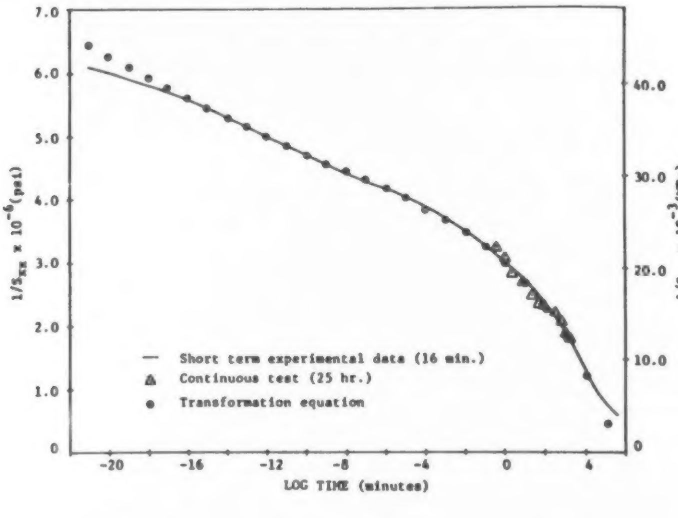


FIG. 46: MASTER CURVE OF THE RECIPROCAL OF REDUCED COMPLIANCE, $1/S_{xx}$, OF $[30°]_{8s}$ LAMINATE AT 180°C[328]

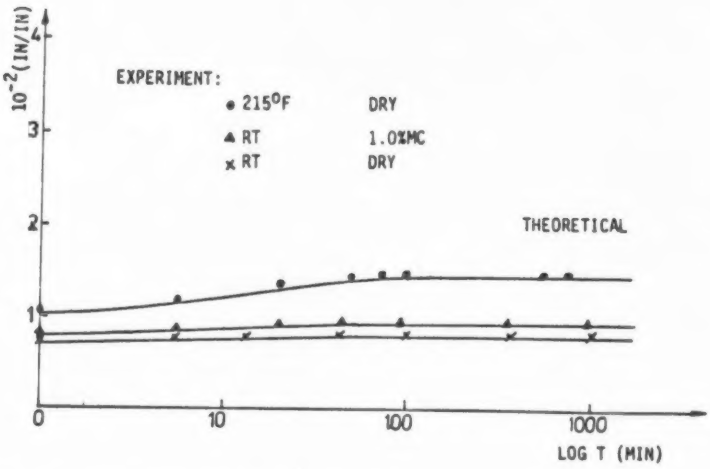


FIG. 47: COMPARISON OF THEORETICAL AND EXPERIMENTAL CREEP FOR QUASI-ISOTROPIC LAMINATE (14000 psi = 97 MPa)[301]

APPENDIX A — BIBLIOGRAPHY

ENVIRONMENTAL EFFECTS ON COMPOSITE MATERIALS

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AUTHOR(S)/AUTEUR(S)

J.P. Komorowski
5

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Aeronautical Note
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CORPORATE AUTHOR/PERFORMING AGENCY/AUTEUR D'ENTREPRISE/AGENCE D'EXÉCUTION

National Research Council Canada
National Aeronautical Establishment Structures and Materials Laboratory
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DESCRIPTORS (KEY WORDS)/MOTS-CLÉS

1. Fibre Composites

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SUMMARY/SOMMAIRE

This is part IV of a series of literature reviews on hygrothermal effects on polymer matrix composite materials. It contains a review of papers on mechanical properties as measured in fatigue, creep or stress relaxation tests with variations in temperature and humidity accounted for in the results.

The other parts of the review are:

Part I: Moisture and Thermal Diffusion
Part II: Physical Properties
Part III: Mechanical Properties 1
Part V: Composite Structures and Joints
Part VI: Numerical and Analytical Solutions
Part VII: Summary of Conclusions and Recommendations

A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.
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